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Manual for the Certification of Laboratories Analyzing Drinking Water Criteria and Procedures Quality Assurance

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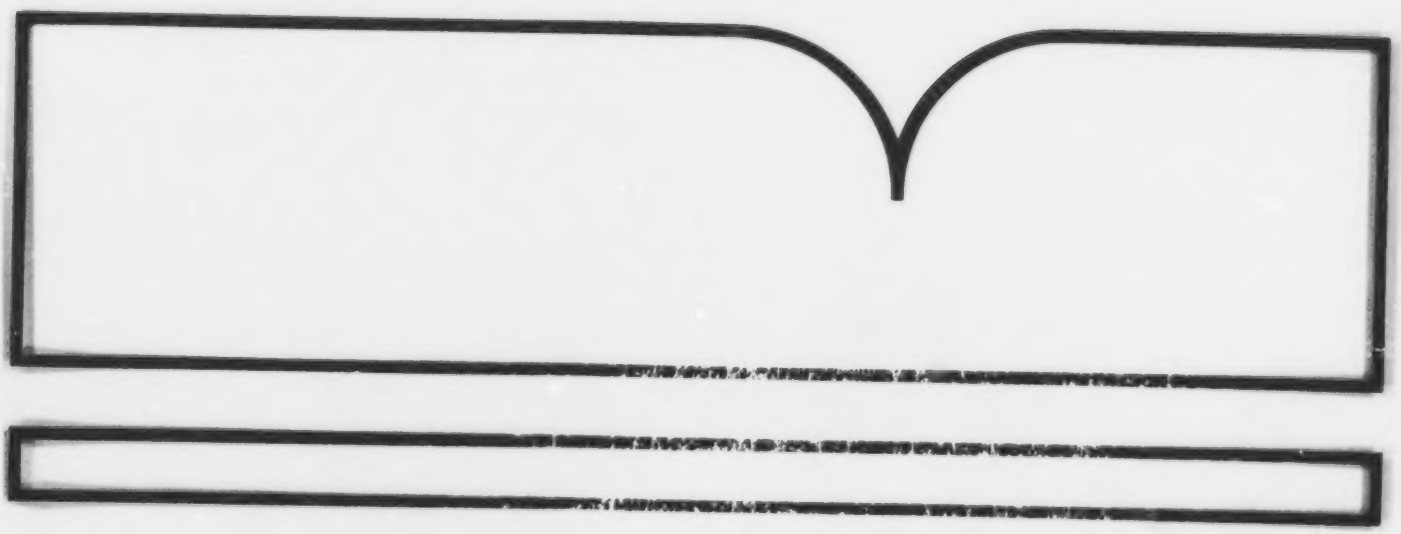

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16. ABSTRACT

This document is a revision of the Manual for the Interim Certification of Laboratories Involved in Analyzing Public Drinking Water Supplies (EPA 600/8-78-008) It was prepared in an effort to update and impove technical criteria and implementation strategy in light of newly approved methodology and over four years of experience with the program. The technical criteria for chemistry, microbiology, and radiochemistry have been updated, clarified, and reorganized, but are similar in substance to the earlier edition cited above. In addition, the program now includes criteria and procedures for down-grading a laboratory's certification status. Like the previous edition, this program is not regulatory in nature (except for methodology), but rather is guidance describing the minimum procedures and criteria for assuring data validity.

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Disclaimer

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Criteria and Procedures Quality Assurance

Prepared by
The Drinking Water Laboratory Certification Implementation
Work Group

Supersedes EPA 600/8-78-008, May 1978, entitled *Manual for the Interim Certification of Laboratories Involved in Analyzing Public Drinking Water Supplies*

Preface

his document is a revision of the **Manual r the Interim Certification of Laboraries Involved in Analyzing Public Drinkg Water Supplies,** EPA 600/8-78-008. It as prepared by the Drinking Water Labotory Certification Implementation Work roup in an effort to update and improve chnical criteria and implementation rategy in light of newly approved methodogy and over four years of experience with e program. The technical criteria for emistry, microbiology, and radiochemistry ave been updated, clarified, and reorganed, but are similar in substance to the arlier edition of the manual. Implementan policy is very similar to the earlier lition, but "Interim Approval" status has en eliminated and the term "Interim rtification" has been changed to "Certifiion." In addition, the program now inudes criteria and procedures for downading a laboratory's certification status. omments from the Regions and States ere solicited and considered in the prepation of this revised manual. These inuded recommendations from a workshop ld in June 1981, in which all Regions and ates were invited to participate and share eir views with respect to both the techni l criteria and implementation policy. most all majority views at that workshop ere incorporated into this manual.

There is a U.S. EPA quality assurance ogram which covers all activities relating data collection, processing, and reportg. This is managed by the Quality Assurnce Management Staff (QAMS), the Office Research and Development. The portion the QAMS program applicable to laboraries conducting drinking water analyses is dicated in Chapter III (Quality Assurance an.)

Like the previous edition, this program is t regulatory in nature (except for methodogy), but rather is guidance describing the inimum procedures and criteria for assurg data validity.

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Chapter I

Introduction

Public water systems serving 25 persons or more must conform with the provisions of the Safe Drinking Water Act, as amended (42 U.S.C. S 300f **et seq.**) and the requirements of the National Interim Primary Drinking Water Regulations (NIPDWR) 40 CFR 141 and 142). Section 1401 (1) (D) of the Act defines a primary drinking water regulation as consisting of "criteria and procedures . . . including quality control and testing procedures to insure compliance. . . ."

The Regulations at 40 CFR 141.28 require that all testing for compliance purposes, except for turbidity, free chlorine residual, temperature, and pH, be performed by laboratories approved by either the U.S. Environmental Protection Agency U.S. EPA) or those States with primary enforcement responsibility (primacy). This manual is intended to assist in the implementation of Section 141.28 by providing a mechanism for the evaluation of these laboratories in order to help assure the validity of data generated.

The U.S. EPA program extends to its Regional laboratories, principal State and Territorial laboratories in primacy States, laboratories on Federal Indian reservations, and laboratories in States without primacy which perform analyses under the Safe Drinking Water Act.

Primacy States must have a certification program for local laboratories if all analyses are not performed in principal State laboratories (see Table I-1), and should adopt provisions at least as stringent as those in this manual for certifying these laboratories.

The certification program operates as follows: U.S. EPA's Environmental Monitoring and Support Laboratory in Cincinnati, Ohio (EMSL-CI), is responsible for determining the certification status of U.S. EPA Regional laboratories for microbiology and for chemistry. The Environmental Monitoring Systems Laboratory in Las Vegas (EMSL-LV) has this responsibility for radiochemistry. Regional certification officers determine the certification status of principal State laboratories in primacy States and are also responsible for local laboratories in non-primacy States. Evaluations of State laboratories for radiochemistry are conducted by EMSL-LV, except where the Regions have this capability.

Primacy States with certification programs are responsible for certifying local laboratories. Regional laboratories and principal State laboratories must successfully analyze a complete set of unknown performance evaluation (PE) samples annually from EMSL-CI (EMSL-LV, where applicable) and pass an on-site evaluation every three years. The criteria given herein will be used at the on-site evaluation.

Chapter II describes the responsibilities of each of the U.S. EPA organizations for this certification program. Chapter III describes how the program will be operated. Chapters IV, V, and VI cover the technical criteria for chemistry, microbiology, and radiochemistry, respectively, used during an on-site evaluation of a laboratory. The technical criteria for each of Chapters IV, V, and VI are divided into two sections: Critical Elements for Certification and Recommended Practices. The first section details items which are essential for the certification of laboratories performing drinking water analyses under the Safe Drinking Water Act. The second section contains items which are highly recommended for good laboratory operations. Only the first section will be used to determine certification status. Evaluation forms are also included in Chapters IV, V, and VI.

The appendices include recommended chain-of-custody procedures, a recommended protocol and format for conducting on-site laboratory evaluations, which may be used by the evaluators, and abbreviations.

All specifications, whether critical or recommended, are guidance, except for those methods and procedures specified in the NIPDWR (see 40 CFR, Part 141) as mandatory. Laboratories wishing to use an alternative method to those prescribed must first obtain U.S. EPA approval in accordance with the established procedure outlined in Chapter III, Alternate Analytical Techniques. The U.S. EPA may approve alternate test procedures on either a case-by-case basis or on a nationwide basis.

Table I-1. Requirements of Primacy

To obtain and maintain primary enforcement responsibility (primacy), a State must conform to the following provisions of 40 CFR 142.10 (b):

(3) The establishment and maintenance of a State program for the certification of laboratories conducting analytical measurements of drinking water contaminants pursuant to the requirements of the State primary drinking water regulations including the designation by the State of a laboratory officer, or officers, certified by the Administrator, as the official(s) responsible for the State's certification program. The requirements of this paragraph may be waived by the Administrator for any State where all analytical measurements required by the State's primary drinking water regulations are conducted at laboratories operated by the State and certified by the Agency. Until such time as the Agency establishes a National quality assurance program for laboratory certification the State shall maintain an interim program for the purpose of approving those laboratories from which the required analytical measurements will be acceptable.

(4) Assurance of the availability to the State of laboratory facilities certified by the Administrator and capable of performing analytical measurements of all contaminants specified in the State primary drinking water regulations. . . .

Chapter II

Responsibilities

The success of the laboratory certification program depends upon cooperation among the organizations responsible for its implementation. This is achieved primarily through the Drinking Water Laboratory Certification Implementation Work Group (DWLCI). Within the Agency, primary responsibilities for laboratory certification are shared by the Office of Drinking Water (ODW), the Office of Research and Development (ORD), and the U.S. EPA Regional Offices.

Office of Drinking Water

ODW is responsible for supervising the national certification program for laboratories analyzing drinking water samples and for implementing the Safe Drinking Water Act, including the preparation of regulations and standards.

Office of Research and Development

The EMSLs at Cincinnati and Las Vegas share responsibility with ODW in developing and implementing the laboratory certification program.

EMSL-CI is the lead organization for operational control of the national certification program for laboratories performing chemical and microbiological analyses. EMSL-CI:

- Reviews Regional certification programs and conducts on-site evaluations of all Regional laboratories every three years;
- Prepares and distributes unknown PE samples and known quality control (QC) samples for all regulated chemical and microbiological contaminants;
- Conducts water supply performance evaluation studies at least annually for all Regional and principal State laboratories, and laboratories in non-primacy States wishing certification. Other laboratories may participate;
- Evaluates the resources and personnel available in each U.S. EPA Region to carry out the certification program;
- Participates in all DWLCI Work Group activities;
- Develops and participates in training courses to support the certification program (if resources are available); and
- Provides technical assistance, as required.

EMSL-LV is the lead organization for operational control of the certification program for laboratories performing radiochemical analyses. Its duties correspond with those described for EMSL-CI. In addition, at the request of a Region, EMSL-LV is responsible for conducting on-site inspections for radiochemistry capability of principal State laboratory systems and, if resources are available, other laboratories not covered by State certification programs. In these cases, EMSL-LV will report the results of its inspection to the responsible Regional Administrator, who will have final authority to determine certification status.

U.S. EPA Regional Offices

The ten Regions are responsible for overseeing progress of the certification program in the States. The Regions:

- Monitor the adequacy of State programs for certifying laboratories;
- Evaluate the certification status of principal State laboratories and laboratories in non-primacy States. This includes an on-site inspection at least once every three years. The Regional Administrator is certifying authority;
- Coordinate EMSL water supply performance evaluation studies with laboratories in the Region;
- Participate in the annual review of certification programs and performance evaluation reports;
- Provide technical assistance to U.S. EPA-certified drinking water laboratories, as needed;
- Operate the certification program in non-primacy States;
- Insure that the Regional laboratory continually maintains its capability of meeting all certification criteria; and
- Maintain a current list of the certification status of all laboratories in the Region certified by U.S. EPA or the States.

In non-primacy States, the Region, in addition to its laboratory certification duties, has administrative, enforcement, and local laboratory certification responsibilities normally performed by the State. Some of these duties may be performed by the State, but the Region must retain responsibility for the on-site evaluation of the designated principal State laboratory. Local laboratories may be evaluated by the Region or under a Region-approved program carried out by a designated principal State laboratory. In either case, this manual shall provide the basis for the on-site evaluation.

Drinking Water Laboratory Certification Implementation Work Group

The DWLCI Work Group was established to oversee the operation of the national certification program for drinking water laboratories. This group advises ODW and includes representatives from ODW, ORD (EMSL-CI, EMSL-LV, MERL and QAMS), Office of Water Enforcement and Permits, Regional Offices, and States. The DWLCI Work Group:

- Monitors the certification program and recommends technical and administrative revisions to ODW as dictated by experience or updated information;
- Develops guidance and responds to questions and comments from the States and Regions;
- Develops technical and administrative criteria to support additional certification needs imposed by future regulations;
- Ascertains laboratory availability and capability for future regulatory activities; and
- Makes recommendations to ODW on resources needed to implement the certification program.

Chapter III

Implementation of Certification Program

Certification of Regional Laboratories and Programs

EMSL-CI is responsible for certifying the Regional laboratory, if one exists, for microbiological and chemical analyses, and for approving the Regional program for certifying other laboratories for these parameters. EMSL-LV has similar responsibilities for a Region having radiochemistry capability. The Regional certification program must be approved before a Region can exercise its authority to certify other laboratories.

Certification of Laboratories

Regional laboratories analyzing potable water samples under the Safe Drinking Water Act must meet the minimum criteria specified in this manual, pass an on-site inspection at least once every three years, and satisfactorily analyze an annual set of PE samples. For those Regions certified for radiochemistry, satisfactory performance on two cross check samples per year is also necessary.

Individual(s) Responsible for Certification Program

The U.S. EPA Region must designate an individual(s) to coordinate drinking water certification activities. This individual(s) must be experienced in quality assurance; hold an advanced degree or have equivalent experience in microbiology, chemistry, or radiochemistry; and have sufficient administrative and technical stature to be considered a peer of the director of the principal State laboratory.

On-Site Evaluation Team

One or more teams must be established by the Region to evaluate a laboratory in microbiology and chemistry. Team members must be experienced professionals, holding at least a bachelor's degree (or equivalent education and experience) in the specific discipline being evaluated. Team members must participate in training activities as specified by EMSL-CI.

Development of Regional Plans for Certifying Local Laboratories in Non-Primacy States

Regions are required to develop plans for certifying local drinking water laboratories in non-primacy States. Written plans should include the following:

- Certification official;
- Types and numbers of laboratories;
- Analyses to be examined;
- Schedule for on-site evaluations; and
- Plans for providing technical assistance to laboratories in need of upgrading.

Certification of State Laboratories

The principal State laboratory system must have the capability to analyze every parameter included in the drinking water regulations (40 CFR 142.10(b)(4)); however, an individual laboratory which is part of a principal State laboratory system may be certified for only one, several, or all the cited analyses. If a principal State laboratory contracts with another laboratory, including a laboratory outside the State, to assume the lead role in analyzing a regulated parameter (e.g., radiochemical contaminants), that contract laboratory will, for the purposes of this manual, be considered part of the principal State laboratory system. In this case, the contract laboratory must be certified either by U.S. EPA or by the State in which the laboratory is located for the contaminants of interest. In the latter case, the State must have primacy.

The certification process for a principal State laboratory or a local laboratory in a non-primacy State will begin when the laboratory director makes a formal request to the Region. This application may result from the following:

- A request for first-time certification for microbiology, chemistry and/or radiochemistry;
- A request for certification to analyze additional or newly regulated parameters;
- A request to renew a laboratory's certification status after three years; and
- A request to reapply for certification after correction of deficiences which resulted in the downgrading/revocation of certification status.

The Region should respond to a formal application for any of the requests within 30 days, and a mutually agreeable date and time should be set for the on-site laboratory evaluation. The recommended protocol for conducting these evaluations is given in Appendix B. For certification, a laboratory must pass an on-site inspection and satisfactorily analyze performance evaluation samples for those parameters for which it requests certification.

After the on-site visit and the review of PE sample results, the Region can classify the laboratory for each type of analysis according to the following ranking scheme:

- **Certified**—a laboratory that meets the minimum requirements of this manual. The certification shall be valid for up to three years.
- **Provisionally Certified**—a laboratory which has deficiencies but can still produce valid data.
- **Not Certified**—a laboratory possessing major deficiencies and, in the opinion of the Regional Administrator, cannot consistently produce valid data.

In the case of laboratories classified as Provisionally Certified, up to one year will be permitted for correction of the deficiencies. A one-time extension of no more than 6 months may be considered by the Region as long as the laboratory is making "good faith" progress in the resolution of its deficiencies and the continued provisional status does not impact the generation of valid data. A Provisionally Certified laboratory may analyze drinking water samples for compliance purposes. In no case should provisional certification be given if the evaluation team believes that the laboratory lacks the capability of performing the analysis within specified limits. Once deficiencies have been corrected to the Regional Administrator's satisfaction, the latter should upgrade the laboratory to Certified status.

For laboratories requesting first-time certification or certification to analyze additional or newly-regulated parameters, the Region, at its discretion, may administratively grant a laboratory Provisionally Certified status, pending an on-site evaluation. It is granted only when the Region judges that the laboratory has both the appropriate instrumentation and trained personnel to perform the analyses, and the laboratory has satisfactorily analyzed PE samples for the contaminants in question.

For those Regions lacking the expertise required to certify laboratories in radiochemistry, EMSL-LV will conduct on-site inspections.

Certification of Local Laboratories

For the purposes of this document, local laboratories include any State, county, municipal, utility, Federal, or commercial laboratory, but excludes principal State laboratories and U.S. EPA Regional laboratories. In non-primacy States, the Regions will certify local laboratories, using the criteria and policies in this manual.

Only primacy States where not all drinking water analyses are conducted at State-operated laboratories are required to establish a certification program for local laboratories (see 40 CFR 142.10(b), Table I-1.). All States, however, are encouraged to develop such programs. Certification must be based upon criteria contained in this manual or State-developed equivalents at least as stringent as those herein. Those States required by regulation to develop a certification program must appoint a laboratory certification officer(s), certified by the Region, as the official(s) responsible for the State program.

The principal State laboratory system must have the technical capability to analyze for all regulated contaminants. If the principal State laboratory has the resources to perform 100% of the analyses for one contaminant (e.g., lead), but does not have adequate resources to perform 100% of the analyses for another contaminant (e.g., can only analyze 20% of all total coliform samples), then the State certification program need only include certification criteria for contaminants for which the State will not be analyzing 100% of the samples.

Federal facilities must comply with all Federal, State and local requirements with respect to the Safe Drinking Water Act. For the purpose of certification, Federal laboratories in which routine monitoring of public drinking water supplies is conducted are to be considered local laboratories. The agency with primary enforcement authority, either the State or the Region, will be responsible for carrying out certification activities. If requested by the State, the Region may conduct on-site evaluations of Federal laboratories in that State. U.S. EPA will have primary enforcement authority over any facilities on Federal Indian lands.

Other Considerations for Laboratory Certification

Quality Assurance Plan

It is essential that all laboratories analyzing drinking water compliance samples adhere to defined quality assurance procedures. This is to insure that routinely generated analytical data are scientifically valid and defensible and are of known and acceptable precision and accuracy. To accomplish these goals, each laboratory should prepare a written description of its quality assurance activities (a QA Plan). The following items should be addressed in each QA plan:

1 Sampling procedures.

2 Sample handling procedures.

— specify procedures used to maintain integrity of all samples, i.e., tracking samples from receipt by laboratory through analysis to disposal.

— samples likely to be the basis for an enforcement action may require special safeguards (see Chain-of-Custody procedures).

3 Instrument or equipment calibration procedures and frequency of their use.

4 Analytical procedures.

5 Data reduction, validation and reporting.

— data reduction: conversion of raw data to μg/L, picocuries/L, coliforms/100 mL, etc.

— validation: includes insuring accuracy of data transcription and calculations.

— reporting: includes procedures and format for reporting data to utilities, State officials, and U.S. EPA.

6 Types of internal quality control (QC) checks and frequency of their use.

— may include preparation of calibration curves, instrument calibrations, replicate analyses, use of EMSL-provided QC samples or calibration standards and use of QC charts.*

7 Preventive maintenance procedures and schedules.

8 Specific routine procedures used to determine data precision and accuracy for each contaminant measured.

— precision is based on the results of replicate analyses.

— accuracy is normally determined by comparison of results with "known" concentrations in reagent water standards and by analyses of water matrix samples before and after adding a known contaminant "spike".

9 Corrective action contingencies.

— response to obtaining unacceptable results from analysis of PE samples and from internal QC checks.

The QA plan may consist of already available standard operating procedures (SOPs) which are approved by the laboratory director and which address the listed items, or may be a separately prepared QA document. Documentation for many of the listed QA plan items can be by reference to appropriate sections of this manual, the laboratory's SOPs or to other literature (e.g., **Standard Methods for the Examination of Water and Wastewater**).

If a particular listed item is not relevant, the QA plan should state this and provide a brief explanation (e.g., some laboratories never collect samples and thus have no need to describe sampling procedures). A laboratory QA plan should be concise but responsive to the above-listed items (a maximum of five pages is suggested). Minimizing paperwork while improving dependability and quality of data are the intended goals.

Chain-of-Custody Procedures

Certified laboratories which may be requested to process a sample for possible legal action against a supplier must have a chain-of-custody procedure available. An example of such a procedure is found in Appendix A.

Requirements for Maintaining Certification Status

Periodic Performance Evaluation (PE) Samples

Certified drinking water laboratories must satisfactorily analyze PE samples on an annual basis for each chemical, radiochemical, or microbiological parameter (when available) for which certification has been granted. Results must be within the acceptance limits established by U.S. EPA for each analysis. To maintain certification in radiochemistry, the laboratory must satisfactorily analyze two cross check samples per year in addition to the annual set of PE samples.

*QC chart for chemistry is explained in **Handbook for Analytical Quality Control in Water and Wastewater Laboratories**, EPA-600/4-79-019, March 1979. QC chart for radiochemistry is explained in **Handbook for Analytical Quality Control in Radioanalytical Laboratories**, EPA-600/7-77-088, August 1977.

Methodology

Laboratories must use methodologies sanctioned by the drinking water regulations (40 CFR 141.21 – 141.25) or otherwise approved by U.S. EPA for compliance with the Safe Drinking Water Act.

Notification of Certifying Authority (CA) for Major Changes

Laboratories must notify the appropriate CA (Regional Administrator or the appropriate EMSL), in writing, within 30 days of major changes in personnel, equipment, or laboratory location which might impair analytical capability. A major change in personnel is defined as the loss or replacement of the laboratory supervisor or a situation in which a trained and experienced analyst is no longer available to analyze a particular parameter for which certification has been granted. The CA will discuss the situation with the laboratory supervisor and establish a schedule for the laboratory to rectify deficiencies.

On-Site Evaluation

The CA must be satisfied that a laboratory is maintaining the required standard of quality for certification. Normally, this will be based upon recommendation of a U.S. EPA on-site evaluation conducted at least every three years.

Criteria and Procedures for Downgrading/Revoking Certification Status

Criteria for Downgrading Certification Status

A laboratory may be downgraded to a Provisionally Certified status for a particular contaminant analysis for any of the following reasons:

1. Failure to analyze a PE sample (or an EMSL-LV cross check sample) within the acceptance limits established by U.S. EPA. If more than one concentration of a particular contaminant is provided, the laboratory must satisfactorily analyze all concentrations, except where otherwise stated. After downgrading to a Provisionally Certified status, a laboratory may request that U.S EPA provide QC samples (standard solution samples for radiochemical contaminants) and technical assistance to help identify and resolve the problem. Provisionally Certified status will continue until the laboratory's analysis of a follow-up PE sample (or EMSL-LV cross check sample) produces data within the acceptance limits established by U.S. EPA.

2. Failure of a certified laboratory to notify the CA within 30 days of major changes in personnel, equipment, or laboratory location which might impair analytical capability.

3. Failure to satisfy the CA that the laboratory is maintaining the required standard of quality, based upon an U.S. EPA on-site evaluation.

During the provisional status period, which may last for up to one year with a possible six month extension, the laboratory may continue to analyze samples for compliance purposes until it resolves its difficulties. It must, however, notify its clients of its downgraded status.

Criteria for Revoking Certification Status

A laboratory may be downgraded from Certified or Provisionally Certified status to a Not Certified classification for a particular contaminant analysis for the following reasons:

1. Failure to analyze an initial and follow-up PE sample (or EMSL-LV cross check sample) for a particular contaminant within the acceptance limits established by U.S. EPA.

2. Failure to correct identified deviations (including continued use of unapproved methods and equipment) by the time specified by the CA.

3. Submission of a PE sample to another laboratory for analysis and reporting data as its own.

4. Falsification of data or other deceptive practices.

Procedures for Revocation

The CA will notify the laboratory in writing (registered or certified mail) of the intent to revoke certification. If the laboratory wishes to challenge this decision, a notice of appeal must be submitted in writing to the CA within 30 days of receipt of the notice of intent to revoke certification. If no notice of appeal is so filed, certification will be revoked.

The notice of appeal must be supported with an explanation of the reasons for the challenge and must be signed by a responsible official from the laboratory such as the president/owner for a commercial laboratory, or the laboratory supervisor in the case of a municipal laboratory.

Within 60 days of receipt of the appeal, the CA will make a decision and notify the laboratory in writing. Denial of the appeal results in immediate revocation of the laboratory's certification. The CA will request the laboratory to notify its clients of its status in writing, and to submit verification that this has been accomplished. Once certification is revoked, a laboratory may not analyze drinking water samples for compliance purposes until its certification has been reinstated.

If the appeal is determined to be valid, the CA will take appropriate measures to re-evaluate the facility and to issue to the laboratory within 60 days a written decision on its certification status.

Reinstatement of Certification

Certification will be reinstated when and if the laboratory can demonstrate to the CA's satisfaction that the deficiencies which produced Provisionally Certified status or revocation have been corrected. This may include an on-site evaluation, a successful analysis of samples on the next regularly scheduled EMSL water supply performance evaluation study, or any other measure the CA deems appropriate.

Reciprocity

Reciprocity, which is defined as mutually acceptable certification among Regions and States, is endorsed by U.S. EPA as a highly desirable element in the certification program for drinking water laboratories. States are encouraged to adopt provisions in their laws and regulations to permit it. States may request U.S. EPA to arbitrate disputes involving reciprocity. Data from U.S. EPA-certified laboratories will be acceptable under the Safe Drinking Water Act in jurisdictions where U.S. EPA has primary enforcement responsibility.

Training

Training functions are no longer provided in support of U.S. EPA's laboratory certification program. Training, however, is an integral part of the laboratory certification process for: a) personnel responsible for certifying laboratories either on behalf of the Regional Office or a primacy State; b) the laboratory analysts responsible for microbiological, chemical, and radiochemical measurements. Mechanisms for providing adequate training should be examined by primacy agencies or other groups.

Technical Services

Reference Samples

There are four types of EMSL reference samples: calibration standards, quality control (QC), performance evaluation (PE), and intercomparison cross check samples. EMSL-CI provides QC and PE samples for all regulated chemical and microbiological contaminants and residual chlorine, and in addition, provides calibration standards for trace organic chemicals. EMSL-LV provides calibration standards, PE samples, and intercomparison cross check samples for all regulated radiochemical contaminants.

QC samples and standards are provided on request as part of a laboratory's own quality assurance activities (see section on Quality Assurance Plan). Contaminant concentrations are furnished. They serve as independent checks on reagents, instruments, and analytical techniques; as an aid for testing or training analysts; or for determining within laboratory precision and accuracy. Although no certification or other formal U.S. EPA evaluation functions result from using these samples, their routine use is considered fundamental to a proper laboratory QA plan.

EMSL-CI and EMSL-LV conduct periodic water supply performance evaluation studies using PE samples as a requirement for certification. In contrast to QC samples and calibration standards, contaminant concentrations are not furnished before analysis. Laboratories should request PE samples through the appropriate Regional Office for chemistry and microbiology, or EMSL-LV for radiochemistry.*

At the conclusion of each study, the EMSLs prepare individual reports for each laboratory and provide them to the participants. The certifying authority reviews unacceptable data with the laboratory to identify and resolve problems. QC samples and calibration standards are useful for this purpose. Once problems are corrected, the laboratory must analyze a second series of PE samples for problem parameters during a follow-up EMSL study.

In addition to the annual PE sample requirement, EMSL-LV also requires satisfactory performance in two intercomparison cross check studies per year. Intercomparison cross check samples differ from PE samples in that the former contain only one or two radionuclides (e.g., radium-226 and radium-228), while PE samples for radiochemistry are complex mixtures of alpha, beta, and photon-emitting radionuclides. In neither case are contaminant concentrations furnished to the laboratory until after completion of the study.

Early Warning System for Problems With Test Supplies and Equipment

A voluntary national system has been established to (1) identify potential problems with chemical and microbiological test materials and equipment; (2) notify the U.S. EPA, manufacturers, and users of these problems; and (3) encourage improvements and tighter quality control over the products. The problems are concerned with performance, QA, specification, design, and labeling of microbiological media and membrane filters, chemical reagents, and other supplies, equipment, and instrumentation used in microbiological and chemical analyses of drinking water. EMSL-CI has the responsibility for maintaining a QA program on methodologies and test materials, and serves as the focal point for identifying and reporting significant problems with such materials to the users and the manufacturers. The following protocol is used:

1. State and local drinking water laboratories or Regional staff members should report microbiological and chemical problems by phone or in writing to the Microbiology Section (513-684-7319) or the Physical and Chemical Methods Branch (513-684-7306), respectively, of EMSL-CI, USEPA, 26 W. St. Clair St., Cincinnati, Ohio 45268. Forms for written reports are provided in Figures III-1 and III-2. A copy of the report should be sent to the QA Officer in the appropriate Region.

2. EMSL-CI will record the details of the problem, including name and location of the reporting laboratory; product type, manufacturer, lot/catalog/model numbers and date received; description of the problem; specific observations; method of preparation, and length and conditions of storage for media or reagents; and data documenting unacceptable test results.

3. EMSL-CI will then describe the reported problem to the manufacturer, obtain manufacturing and QA data, and discuss its significance. Corrections or changes by the manufacturer will be encouraged.

4. Based on the results of discussions with the reporter(s) of the problem and the manufacturer, EMSL-CI will alert the Regional QA Officers of *possible* problems with the product. The QA Officers will alert the appropriate U.S. EPA and State personnel. This system is not intended to label the media, reagents, or other materials as unacceptable, but rather to alert water laboratories that a problem may exist and to determine if similar problems have been observed elsewhere.

5. If multiple reports of the same problem are received, EMSL-CI will inform the manufacturer of a potentially broad-scope problem and request samples from reporting laboratories for testing.

6. If the product is unsatisfactory in these tests, EMSL-CI will notify the manufacturer and the Regional QA Officers who, in turn, will notify the Regional, State, and local authorities.

Alternate Analytical Techniques

Section 141.27 of the NIPDWR permits approval of alternate analytical techniques. Such a technique, also known as an alternate test procedure (ATP), shall be accepted "... only if it is substantially equivalent to the prescribed test in both precision and accuracy as it relates to the determination of compliance with any maximum contaminant level."

EMSL-CI, through its Equivalency Staff, is responsible for and provides coordination of a program to determine the acceptability of proposed techniques, and makes recommendations for approval or denial to the appropriate authority. Applications for approval of an ATP may be made on a limited or a nationwide basis. Requests for limited use approvals are submitted to the appropriate Regional Administrator or designee who, after receiving recommendations from the Director of EMSL-CI and the Director of ODW, has the final authority to approve requests. Requests for nationwide use approvals should be forwarded directly to the Director of EMSL-CI, who, after review by the Equivalency Staff, will provide recommendations to the Director of ODW, with whom final authority for approval rests.

Applicants who propose an ATP need to provide a step-by-step procedure, applicable literature citations or other references, and any available comparability data between the proposed ATP and the USEPA-approved test procedure for the same contaminant.

In the case of new techniques where precision and accuracy data may not be available to support an application, the applicant for a limited use ATP will be asked to provide comparability data from the analysis of samples collected from one to five water supply systems most representative of those routinely analyzed. From each of these systems, three samples, in which the concentrations range from the limit of detection (LD) to the maximum contaminant level (MCL), should be collected and each sample analyzed eight times, four times each, by the proposed technique and the U.S. EPA-approved test procedure. Samples can be spiked as necessary to cover the concentration range between the LD and MCL. Applicants for a nationwide ATP may also be asked to provide comparability data. For each of a minimum of five sources, six samples should be collected and analyzed eight times, four times by each technique. The water supply sources selected must be dispersed geographically throughout the United States. Samples may also be spiked as necessary to cover the concentration range between the LD and the MCL.

The EMSL-CI Equivalency Staff will apply a series of proven statistical techniques to the data submitted with all applications to determine equivalency between the proposed and the approved techniques, and submit recommendations to approval authorities.

*EMSL-LV address is U.S. EPA/EMSL, P.O. Box 15027, Las Vegas, Nevada 89114

Figure III-1

Report of Problem With Microbiological Supplies or Equipment

Product* _____ Date _____

Manufacturer _____

Address _____

Date Received _____ Expiration Date _____

Lot No. _____ Cat. No. _____ Model No. _____

Description of Problem: ** _____

_____

_____

_____

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_____

_____

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_____

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_____

_____

_____

_____

_____

Name _____ Phone No. _____
(Person Reporting)

Laboratory/Facility _____

Address _____

_____

*Membrane Filters, Microbiological Media, Reagents, Portable Incubators, Waterbaths, etc.

**Information should include the length and condition of storage, and the method of preparation for media and reagents. Specific observations, quality control checks, and data that document unacceptable results are useful in describing the problem.

Send to: Microbiology Section, EMSL-CI, U.S. EPA, 26 W. St. Clair St., Cincinnati, OH 45268, or phone (513) 684-7319.

Figure III-2

Report of Problem With Chemical Supplies or Equipment

Product* _____ Date _____

Manufacturer _____

Address _____

Date Received _____ Expiration Date _____

Lot No. _____ Cat. No. _____ Model No. _____

Description of Problem: ** _____

Name _____ Phone No. _____
(Person Reporting)

Laboratory/Facility _____

Address _____

*Chemicals, Prepared Reagents, Instruments, etc.

**Information should include the length and condition of storage, and the method of preparation for reagents. Specific observations, quality control checks, and data that document unacceptable results are useful in describing the problem.

Send to: Physical and Chemical Methods Branch, EMSL-CI, U.S. EPA, 26 W. St. Clair St., Cincinnati, OH 45268, or phone (513) 684-7306.

Chapter IV

Chemistry

Critical Elements for Certification

The technical criteria in this chapter are divided into two sections: Critical Elements for Certification and Recommended Practices. Only the first section will be used to determine certification status.

1. Personnel

Although there are no critical elements for laboratory analysts, laboratory administrators and evaluators should recognize training and experience as essential to the acquisition of valid compliance monitoring data. Recommended minimum standards can be found in Recommended Practices.

2. Laboratory Facilities

There are no critical elements for certification of laboratory facilities for chemistry. Minimum standards are inherent to the instrumentation required to perform the tests. Additional recommendations may be found in Recommended Practices.

3. Laboratory Equipment and Supplies

Only those instruments needed to perform the approved methodology for the contaminants for which the laboratory is being certified are required. Those instruments, however, must meet the following specifications. Additional useful information is available in the document "Guidelines for the Selection of Laboratory Instruments," AWWA No. M 15, American Water Works Association.

3.1 General

3.1.1 Analytical balance: Sensitivity of at least 0.1 mg. The balance must be seated on a steady base to prevent interference due to vibration and should be protected from interference due to air currents.

3.1.2 Magnetic stirrer: Variable speed, with stirring bar coated with inert material.

3.1.3 pH meter: Accuracy, ± 0.05 units. Scale readability, ± 0.1 units. Laboratories purchasing a new pH meter are strongly advised to purchase one capable of functioning with specific ion electrodes. Unit may be line/bench or battery/portable operated.

3.1.4 Conductivity meter: Suitable for checking distilled water quality. Should be readable in ohms or mhos, have a range from 2 ohms to 2 megohms or equivalent micromhos ± 1 percent. Unit may be line/bench or battery/portable operated.

3.1.5 Hot plate: Large or small units with selectable temperature controls for safe heating of laboratory reagents.

3.1.6 Refrigerator: A standard kitchen type domestic, commercial, or laboratory grade refrigerator for storage of aqueous reagents and samples.

3.1.7 Drying oven: Gravity or mechanical convection units with selectable temperature control from room temperature to 180°C (±2°) or higher.

3.1.8 Thermometer: Any good grade mercury-filled centigrade thermometer with 1°C or finer subdivisions calibrated to 180°C or higher.

3.2 Inorganic contaminants

3.2.1 Photometer:

3.2.1.1 Spectrophotometer: Usable wavelength range, 400 to 700 nm. Maximum spectral bandwidth, no more than 20 nm. Wavelength accuracy, ± 2.5 nm. Photometer must be capable of using several sizes and shapes of absorption cells providing a sample path length from approximately 1 to 5 cm.

3.2.1.2 Filter photometer (abridged spectrophotometer): Capable of measuring radiant energy in range of 400 to 700 nm. Relatively broad bands (10 to 75 nm) of this radiant energy are isolated by use of filters or other isolation device at or near the maximum absorption of the colorimetric methods. Photometer should be capable of using several sizes and shapes of absorption cells providing a sample path length varying from approximately 1 to 5 cm.

3.2.2 Specific ion meter: Readable and accurate to ± 1 mV. Unit may be line/bench or battery/portable operated.

3.2.3 Electrodes: pH electrodes, specific ion electrodes and reference electrodes as specified by the individual method.

3.2.4 Stirred water bath: For operation up to 100°C (with gable lid).

3.2.5 Automated analysis systems: Exact equipment used is specified by the individual method; includes:

3.2.5.1 Sampler

3.2.5.2 Proportioning pump

3.2.5.3 Manifold or analytical cartridge

3.2.5.4 Heating bath

3.2.5.5 Heating bath with distillation head

3.2.5.6 Continuous filter

3.2.5.7 Colorimeter with filters

3.2.5.8 Ion selective electrode detector with electrodes

3.2.5.9 Recorder

3.2.6 Arsine generator and absorption system: A Gutzeit generator or equivalent used in conjunction with an absorber tube or assembly.

3.2.7 Atomic absorption spectrophotometer: Single-channel, single- or double-beam instrument having a grating monochromator, photomultiplier detector, adjustable slits, a wavelength range of at least 190 to 800 nm.

3.2.7.1 Readout system: An appropriate readout system that has a response time capable of measuring the atomic absorption signal generated is required. This includes the capability to detect positive interference on the signal from intense non-specific absorption. In furnace analysis a strip chart recorder must be used for verification

of adequate background correction if a CRT video readout or hard copy plotter is not available. The recorder must have a chart width of 10 inches or 25 cm, full scale response time of 0.5 sec. or less, 10- or 100-mV input to match the instrument and variable chart speeds of 5 to 50 cm/min, or equivalent.

3.2.7.2 Fuel and oxidant: Commercial grade acetylene is generally acceptable. Air may be supplied from a compressed air line, a laboratory compressor, or from a cylinder of compressed air. Reagent grade nitrous oxide is also required for certain determinations. Standard, commercially available argon and/or nitrogen are required for furnace work, and hydrogen is required for the flame hydride systems. The supplies of fuel and oxidant shall be maintained at pressures somewhat higher than the controlled operating pressure of the instrument by suitable valves.

3.2.7.3 Burner: The burner recommended by the particular instrument manufacturer and consistent with the approved method should be used. For certain elements the nitrous oxide burner is required.

3.2.7.4 Hollow cathode lamps: Single element lamps are to be preferred but multi-element lamps may be used. Electrodeless discharge lamps may also be used.

3.2.7.5 Graphite furnace: Any furnace device capable of reaching the specified temperatures is satisfactory.

3.2.7.6 Pipets: Microliter with disposable tips. Sizes can range from 5 to 100 microliters as required. Pipet tips which are white in color and do not contain CdS have been found suitable.

3.2.7.7 Background corrector: A background correction system or provision for a subsequent analysis using a non-absorbing line is required for furnace analysis.

3.2.7.8 Separatory funnels: 250 mL, or larger, for extraction with organic solvents.

3.2.7.9 Hydride generation system: Any gaseous hydride system used in conjunction with an atomic absorption spectrophotometer equipped for direct aspiration analysis.

3.2.8 Mercury cold vapor analyzer: Commercially available vapor mercury analyzer can be substituted for the equipment listed below.

3.2.8.1 Absorption cell: Standard 10 cm quartz cell with end windows or 11.5 cm plexiglass cell with an I.D. of 2.5 cm.

3.2.8.2 Air pump: Peristaltic pump with an air flow of 1 L per minute.

3.2.8.3 Flowmeter: Capable of measuring an air flow of 1 L per minute.

3.2.8.4 Spectrophotometer: Atomic absorption spectrophotometer equipped with a mercury hollow cathode lamp.

3.2.8.5 Aeration tube: A straight glass frit having a coarse porosity.

3.2.8.6 Drying unit: A 6 inch drying tube containing 20 grams of magnesium perchlorate or a heating device is required to prevent condensation of moisture.

3.3 Organic Contaminants

3.3.1 Gas chromatograph: A commercial or custom-designed gas chromatograph (GC) with a column oven capable of isothermal temperature control ± 0.2°C to at least 220°C. Additional accessories and specifications are listed below by methodology.

3.3.1.1 Chlorinated hydrocarbons: Equipped with a glass lined injection port suitable for chlorinated hydrocarbon pesticides with a minimum of decomposition, and equipped with either an electron capture, microcoulometric titration, or electrolytic conductivity detector.

3.3.1.2 Chlorophenoxys: Equipped with a glass lined injection port and either an electron capture, microcoulometric titration, or electrolytic conductivity detector.

3.3.1.3 TTHM by purge and trap: Temperature programmable from 45° to 220° at about 8°C/min and equipped with either microcoulometric titration or electrolytic conductivity detector.

3.3.1.4 TTHM by liquid/liquid extraction: Equipped with a linearized (frequency modulated) electron capture detector.

3.3.1.5 TTHM by gas chromatography/mass spectrometry: The gas chromatograph, which must be temperature programmable, should be interfaced to the mass spectrometer with an all-glass enrichment device and an all-glass transfer line. Mass spectral data are to be obtained with electron-impact ionization at a nominal electron energy of 70 eV. The mass spectrometer needs to produce a spectrum that meets all criteria in Table IV-1 when 50 ng or less of p-bromofluorobenzene (BFB) is introduced into the gas chromatograph. An interfaced data system is necessary to acquire, store, reduce and output mass spectral data. The data system needs to be equipped with software to acquire and manipulate data for only a few ions that were selected as characteristic of trihalomethanes and the internal standard (or surrogate compound).

3.3.2 Recorder for gas chromatograph: Strip chart recorder having a chart width of 10 in or 25 cm, a full scale response time of 1 sec. or less, 1-mV (-0.05 to 1.05) signal to match the instrument, and a chart speed of 0.25 to 0.5 in/min or equivalent.

3.3.3 Purge and trap system: A commercial or custom-designed system containing three separate elements. When used with a compatible gas chromatograph, the assembly should be able to detect 0.5 μg/L of each of the individual trihalomethanes and measure them with a reproducibility not to exceed 8% relative standard deviation at 20 μg/L.

TABLE IV-1

p-Bromofluorobenzene Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	96 to 100% of mass 174
177	5 to 9% of mass 176

3.3.3.1 Purging device: Designed for a 5 mL sample volume. Gas inlet disperses finely divided gas bubbles through the sample.

3.3.3.2 Trapping device: Capable of retaining purged trihalomethanes at room temperatures.

3.3.3.3 Desorber assembly: Capable of heating the trapping device to 180°C in one minute with less than 40°C overshoot.

3.3.4 Kuderna-Danish glassware: Sets of tapered glassware, each consisting of a three ball Snyder column, evaporative flask, and calibrated ampul.

3.3.5 Water bath: Electric or steam heated capable of temperature control to within 5°C to 100°C. Concentric ring or other cover is required to support Kuderna-Danish concentrators.

4. General Laboratory Practices

Although laboratory practices are not specified here for laboratory certification, it must be recognized that the generation of valid analytical data is dependent upon proper laboratory practices (see Recommended Practices).

5. Analytical Methodology

5.1 General: All prepackaged kit procedures, other than the DPD Colorimetric Test Kit, are considered alternative analytical techniques, and procedures described under Section 141.27 of the National Interim Primary Drinking Water Regulations (NIPDWR) are to be followed.

5.2 Inorganic contaminants: Table IV-2 shows the approved methodology and references for inorganic chemical contaminants as described in Section 141.23 of the NIPDWR. All other procedures are considered alternataive analytical techniques.

5.3 Organic contaminants: Table IV-3 shows the approved methodology and references for organic chemical contaminants as described in Sections 141.24 and 141.30 of the NIPDWR. All other procedures are considered alternative analytical techniques.

6. Sample Collection, Handling, and Preservation

When the laboratory has been delegated responsibility for sample collection, handling, and preservation, there needs to be strict adherence to correct sampling procedures, complete identification of the sample, and prompt transfer of the sample to the laboratory.

6.1 General

6.1.1 The collector should be trained in sampling procedures and approved by the State regulatory authority or its delegated representative.

6.1.2 The sample needs to be representative of the potable water system. The water tap must be sampled after

Table IV-2

Approved Methodology for Inorganic Contaminants

Contaminant	Methodology	Reference (Method Number) EPA[1]	ASTM[2]	SM[3]	Other
Arsenic	Atomic absorption; furnace technique	206.2	-	-	-
	Atomic absorption; gaseous hydride	206.3	D2972-78B	301A-VII	I-1062-78[4]
	Spectrophotometric, silver diethyldithiocarbamate	206.4	D2972-78A	404A after B(4)	-
Barium	Atomic absorption; direct aspiration	208.1	-	301A-IV	-
	Atomic absorption; furnace technique	208.2	-	-	-
Cadmium	Atomic absorption; direct aspiration	213.1	D3557-78A or B	301A-II or III	-
	Atomic absorption; furnace technique	213.2	-	-	-
Chromium	Atomic absorption; direct aspiration	218.1	D1687-77D	301A-II or III	-
	Atomic absorption; furnace technique	218.2	-	-	-
Fluoride	Colorimetric SPADNS; with distillation	340.1	D1179-72A	414 A and C	-
	Potentiometric ion selective electrode	340.2	D1179-72B	414B	-
	Automated Alizarin fluoride blue; with distillation	340.3	-	603	129-71W[5]
	Automated ion selective electrode	-	-	-	380-75WE[6]
	Zirconium eriochrome cyanine R; with distillation	-	-	-	I-3325-78[4]
Lead	Atomic absorption; direct aspiration	239.1	D3559-78A or B	301A-II or III	-
	Atomic absorption; furnace technique	239.2	-	-	-
Mercury	Manual cold vapor technique	245.1	D3223-79	301A-VI	-
	Automated cold vapor technique	245.2	-	-	-
Nitrate	Colorimetric brucine	352.1	D992-71	419D	-
	Spectrometric; cadmium reduction	353.3	D3867-79B	419C	-
	Automated hydrazine reduction	353.1	-	-	-
	Automated cadmium reduction	353.2	D3867-79A	605	-
	Ion selective electrode	-	-	-	93MM-79[7]
Selenium	Atomic absorption; furnace technique	270.2	-	-	-
	Atomic absorption; gaseous hydride	270.3	D3859-79	301A-VII	I-1667-78[4]
Silver	Atomic absorption; direct aspiration	272.1	-	301A-II	-
	Atomic absorption; furnace technique	272.2	-	-	-

[1] "Methods of Chemical Analysis of Water and Wastes," EPA Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, 45268 (EPA-600/4-79-020), March 1979. Available from ORD Publications, CERI, EPA, Cincinnati, Ohio, 45268. For approved analytical procedures for metals, the technique applicable to total metals must be used.

[2] Annual Book of ASTM Standards, Part 31 Water, American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania, 19103.

[3] "Standard Methods for the Examination of Water and Wastewater," 14th Edition, American Public Health Association, American Water Works Association, Water Pollution Control Federation, 1975.

[4] Techniques of Water Resources Investigation of the United States Geological Survey, Chapter A-1, "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments," Book 5 (1979, Stock #024-001-03177-9). Available from Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

[5] "Fluoride in Water and Wastewater. Industrial Method #129-71 W." Technicon Industrial Systems. Tarrytown, New York, 10591, December 1972.

[6] "Fluoride in Water and Wastewater," Technicon Industrial Systems. Tarrytown, New York, February 1976.

[7] "Methods Manual-93 Series Electrodes." Form 93 MM/9790, pp. 3-6, 1979. Orion Research Incorporated, Cambridge, Mass.

maintaining a steady flow of 2 or 3 minutes to clear service line. The tap must be free of aerator, strainer, hose attachment, or water purification devices.

6.1.3 The sample report form should be completed immediately after collection with location, date and time of collection, collector's name, and any special remarks concerning the sample.

6.2 Inorganic contaminants

6.2.1 The type of sample container and the required preservative for each inorganic chemical contaminant are listed in Table IV-4.

6.2.2 It is essential that all samples be analyzed within the maximum holding times listed in Table IV-4. Where maximum holding times cannot be met, the sample is to be discarded and resampling requested.

6.3 Organic Contaminants

6.3.1 The type of sample container and the required preservative for the organic chemical contaminants are listed in Table IV-5.

6.3.2 When sampling chlorinated waters for TTHM analysis, sodium thiosulfate or sodium sulfite should be added to the empty sample bottles prior to shipping to the sampling site.

6.3.3 The TTHM bottles need to be filled in such a manner that no air bubbles pass through the sample as the bottle is filled. The bottle is to be sealed so that no air bubbles are entrapped in it. The hermetic seal on the sample bottle needs to be maintained until analysis.

6.3.4 It is essential that all samples be analyzed within the maximum holding times listed in table IV-5. Where maximum holding times cannot be met, the sample is to be discarded and resampling requested.

7. Quality Assurance

The critical elements for quality assurance are described below. Additional specifications can be found in the Recommended Practices section.

7.1 General

7.1.1 The laboratory should prepare and follow a written QA plan (see Chapter III, section on QA plans). All quality assurance data should be available for inspection.

7.1.2 It is essential that the laboratory analyze an unknown performance evaluation sample (when available) once per year for all regulated contaminants measured. Results need to be within the control limits established by USEPA for each analysis for which the laboratory wishes to be certified.

7.1.3 A manual of analytical methods should be available to the analysts.

7.1.4 pH meters are calibrated each use period with fresh standard buffers at pH 7.0 and at the pH appropriate for the test being performed.

7.2 Inorganic contaminants

7.2.1 A standard reagent curve composed of a minimum of a reagent blank and three standards covering the concentration range of the samples needs to be prepared. At least one of the standards should be at or below the MCL.

7.2.2 For each day on which analyses are performed, the standard curve needs to be verified by use of at least a laboratory method blank and one standard within the range of the standard curve. Daily checks should be within ± 10 percent of the original curve.

7.2.3 If 20 or more samples per day are analyzed, the working standard curve needs to be verified by running an additional standard within the range of the standard curve every 20 samples. Each check should be within ± 10 percent of original curve.

7.3 Organic contaminants

7.3.1 For each day on which pesticide or phenoxyacid analyses are initiated, or trihalomethane reagent water is prepared, it is essential that a laboratory method blank be analyzed with the same procedures used to analyze samples.

7.3.2 A minimum of three calibration standards should be analyzed each day to calibrate the analytical system. If the laboratory can demonstrate that the instrument response is linear through the origin, this practice can be reduced to one standard per day, providing the response of the standard is within ± 15 percent of previous calibrations.

Table IV-3

Approved Methodology for Organic Contaminants

Contaminant	Methodology	Reference (Method Number or page numbers) EPA[1]	ASTM[2]	SM[3]	USGS[4]
Chlorinated hydrocarbons: endrin lindane methoxychlor toxaphene	Solvent extraction, gas chromatography	pp. 1-19	D3086-79	509A	pp. 24-39
Chlorophenoxys: 2,4-D 2,4,5-TP	Solvent extraction, derivatization, gas chromatography	pp. 20-35	D3478-79	509B	pp. 24-39
Total Trihalomethanes (TTHM)	Purge and trap, gas chromatography	([5])	-	-	-
	Solvent extraction, gas chromatography	([6])	-	-	-
	Gas chromatography/mass spectrometry	([7])	-	-	-

[1] "Methods for Organochlorine Pesticides and Chlorophenoxy Acid Herbicides in Drinking Water and Raw Source Water," Available from ORD Publications, CERI, EPA, Cincinnati, Ohio, 45268.
[2] Annual Book of ASTM Standards, Part 31 Water, American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.
[3] "Standard Methods for the Examination of Water and Wastewater," 14th Edition, American Public Health Association, American Water Works Association, Water Pollution Control Federation, 1975.
[4] Techniques of Water-Resources Investigation of the United States Geological Survey, Chapter A-3, "Methods for Analysis of Organic Substances in Water," Book 5, 1972. Available from Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.
[5] "The Analysis of Trihalomethanes in Finished Waters by the Purge and Trap Method," Method 501.1, EMSL, EPA, Cincinnati, Ohio 45268.
[6] "The Analysis of Trihalomethanes in Drinking Water by Liquid/Liquid Extraction," Method 501.2, EMSL, EPA, Cincinnati, Ohio 45268.
[7] "Measurement of Trihalomethanes in Drinking Water by Gas Chromatography/Mass Spectrometry and Selected Ion Monitoring," Method 501.3, EMSL, EPA, Cincinnati, Ohio 45268.

7.3.3 Each quarter, it is essential that the laboratory analyze certified quality control check standards for each contaminant. If the criteria established by USEPA are not met, corrective action needs to be taken and documented.

7.3.4 It is essential that the laboratory analyze a field blank for trihalomethanes with each sample set. If reportable levels of trihalomethanes are demonstrated to have contaminated the field blank, resampling is essential.

7.3.5 The laboratory is to analyze 10 percent of all samples for TTHM in duplicate. A continuing record of results and subsequent actions taken needs to be maintained.

7.3.6 The laboratory needs to analyze a known TTHM laboratory control standard each day. If errors exceed 20 percent of the true value, all trihalomethane results since the previous successful test are to be considered suspect.

7.3.7 Each time the TTHM analytical system undergoes a major modification or prolonged period of inactivity, the precision of the system needs to be demonstrated by the analysis of replicate laboratory control standards.

7.3.8 It is critical that laboratories that analyze for TTHM by liquid-liquid extraction demonstrate that raw source waters do not contain interferences under the chromatographic conditions selected.

7.3.9 If a mass spectrometer detector is used for TTHM analysis, it is essential that the mass spectrometer performance tests described under equipment specifications using BFB be conducted once during each 8-hour work shift. Records of satisfactory performance and corrective action need to be maintained.

Table IV-4

Sample Collecting, Handling, and Preservation for Inorganic Contaminants[1]

Contaminant	Preservative[2]	Container[3]	Maximum Holding Time[4]
Arsenic	Conc HNO_3 to pH < 2	P or G	6 months
Barium	Conc HNO_3 to pH < 2	P or G	6 months
Cadmium	Conc HNO_3 to pH < 2	P or G	6 months
Chromium	Conc HNO_3 to pH < 2	P or G	6 months
Fluoride	None	P or G	1 month
Lead	Conc HNO_3 to pH < 2	P or G	6 months
Mercury	Conc HNO_3 to pH < 2	G	38 days
		P	14 days
Nitrate			
Chlorinated supplies	Cool, 4°C	P or G	28 days
Non-chlorinated supplies	Conc H_2SO_4 to pH < 2	P or G	14 days
Selenium	Conc HNO_3 to pH < 2	P or G	6 months
Silver	Conc HNO_3 to pH < 2	P or G	6 months

[1]If a laboratory has no control over these factors, the laboratory director must reject any samples not meeting these criteria and so notify the authority requesting the analyses.
[2]If HNO_3 cannot be used because of shipping restrictions, sample may be initially preserved by icing and immediately shipping it to the laboratory. Upon receipt in the laboratory, the sample must be acidified with conc HNO_3 to pH < 2. At time of analysis, sample container should be thoroughly rinsed with 1:1 HNO_3; washings should be added to sample.
[3]P = Plastic, hard or soft; G = Glass, hard or soft.
[4]In all cases, samples should be analyzed as soon after collection as possible.

Table IV-5.

Sample Collection, Handling and Preservation for Organic Contaminants[1]

Contaminant	Preservative	Container	Maximum Holding Time[2]
Chlorinated hydrocarbons	Refrigerate at 4°C as soon as possible after collection	Glass with foil or Teflon-lined cap	14 days[3]
Chlorophenoxys	Refrigerate at 4° as soon as possible after collection	Glass with foil or Teflon-lined cap	7 days[3]
TTHM	Sodium thiosulfate or sodium sulfite	Glass with Teflon-lined septum[4]	28 days

[1]If a laboratory has no control over these factors, it is critical that the laboratory director reject any samples not meeting these criteria and so notify the authority requesting the analyses.
[2]In all cases, samples should be analyzed as soon after collection as possible.
[3]Well-stoppered and refrigerated extracts can be held up to 30 days.
[4]All samples are collected in duplicate.

8. Records and Data Reporting

8.1 Records of chemical analyses are to be kept by the laboratory for not less than 3 years. This includes all raw data, calculations, and quality control data.

8.2 Actual laboratory reports may be kept. However, data, with the exception of compliance check samples as detailed in Section 141.33(b) of the NIPDWR, may be transferred to tabular summaries. The following information should be included:

8.2.1 Date, place, and time of sampling; name of person who collected the sample.

8.2.2 Identification of sample as to whether it is a routine distribution system sample, check sample, raw or process water sample, or other special purpose sample.

8.2.3 Date of receipt of sample and date of analysis.

8.2.4 Laboratory and persons responsible for performing analysis.

8.2.5 Analytical technique/method used.

8.2.6 Results of analysis.

9. Action Response to Laboratory Results

When action response is a designated laboratory responsibility, the proper authority must be promptly notified of non-compliance sample results and a request made for resampling from the same sampling point.

10. Maximum Total Trihalomethane Potential

Laboratories that are engaged in the determination of maximum total trihalomethane potential (MTP) need to meet the following requirements for that test.

10.1 **Method:** Gas chromatography. "Method for the Determination of Maximum Total Trihalomethane Potential – Method 510.1." EMSL, U.S. EPA, Cincinnati, Ohio 45268.

10.2 Sample Container: TTHM sample bottle.

10.3 Supplemental equipment: Constant temperature storage container, water bath or incubator, 25°C or above.

10.4 The laboratory must be certified for TTHM analysis.

11. Sodium and Corrosivity

Measurements for sodium and certain corrosivity characteristics to meet special monitoring regulations are to be performed in an approved laboratory. The critical elements for these tests are described below.

11.1 Methodology – Table IV-6 shows the approved methodology and references for sodium and corrosivity measurements as described in Sections 141.41 and 141.42 of the NIPDWR. All other procedures are considered alternative analytical techniques.

11.2 Additional Criteria

11.2.1 Sodium – Samples may be collected in plastic or glass. Samples are preserved by the addition of conc. HNO_3 to pH < 2; analyses are performed within 6 months. An atomic absorption spectrophotometer or flame photometer is necessary.

11.2.2 Alkalinity – Samples may be collected in plastic or glass, and are preserved by cooling to 4°C; analysis are performed within 14 days.

11.2.3 Calcium – Samples may be collected in plastic or glass, and are preserved by the addition of conc. HNO_3 to pH < 2 and cooling to 4°C; analyses are performed within 6 months.

11.2.4 Chloride – Samples may be collected in plastic or glass. Samples are not preserved; analyses are performed within 7 days. An electronic voltmeter with a glass and silver-silver chloride electrode system is necessary.

11.2.5 Sulfate – Samples may be collected in plastic or glass, and are preserved by cooling to 4°C; analyses are performed within 7 days.

11.2.6 Total filterable residue – Samples may be collected in plastic or glass, and are preserved by cooling to 4°C; analyses are performed within 7 days. Glass or plastic desiccator is necessary.

Recommended Practices

1. Personnel

1.1 Laboratory Director. The following are recommended minimum standards for the laboratory director.

1.1.1 Academic training: Minimum of bachelor of science degree or its equivalent.

1.1.2 Experience: Minimum of 5 years of experience.

1.2 Supervisor. This position may not be necessary in smaller laboratories. The following are recommended minimum standards for the supervisor.

1.2.1 Academic training: Minimum bachelor's degree in chemistry or its equivalent.

1.2.2 Experience: Minimum of 2 years of experience in measurements being considered for certification.

1.3 Analyst for Inorganic Contaminants. The following are recommended minimum standards for the analyst position.

1.3.1 Academic training: Minimum of high school diploma or its equivalent (State certification or licensing may be considered).

1.3.2 Experience: Minimum of 6 months of on-the-job training, under direct supervision of qualified analyst, in measurements being considered for certification.

1.3.3 After 6 months, the analyst must demonstrate acceptable

Table IV-6

Approved Methodology for Free Residual Chlorine, Turbidity, Sodium and Corrosivity Measurements

Measurement	Methodology	Reference (Method Number) EPA[1]	ASTM[2]	SM[3]	Other
Alkalinity	Methyl orange titrimetric or potentiometric	310.1	D1067-70B	403	-
Calcium	EDTA titrimetric	215.2	D1126-67B	306C	-
	Atomic absorption; direct aspiration	215.1	D2576-70	301A-II	-
Chloride	Potentiometric	-	-	408C	-
Corrosivity	Langelier index	-	-	203	-
	Aggressive index	-	-	-	C400-77[4]
Free chlorine residual	Colorimetric or titrimetric DPD	-	-	409E or F	-
	Colorimetric syringaldazine	-	-	-	408G[5]
pH	Potentiometric	150.1	D1293-78A or B	424	-
Sodium	Atomic absorption; direct aspiration	273.1	-	-	-
	Atomic absorption; furnace technique	273.2	-	-	-
	Flame photometric	-	D1428-64A	320A	-
Sulfate	Turbidimetric	375.4	-	427C	-
Temperature	Thermometric	-	-	212	-
Total filterable residue	Gravimetric	160.1	-	208B	-
Turbidity	Nephelometric	180.1	-	214A	-

[1] "Methods of Chemical Analysis of Water and Wastes," EPA Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, 45268 (EPA-600/4-79-020), March 1979. Available from ORD Publications, CERI, EPA, Cincinnati, Ohio, 45268. For approved analytical procedures for metals, the technique applicable to total metals must be used.

[2] Annual Book of ASTM Standards, Part 31 Water, American Society for Testing and Materials, 1916 Race Street, Philadelphia, Penn[illegible]a, 19103.

[3] "Standard Methods for the Examination of Water and Wastewater," 14th Edition, American Public Health Association, American W[illegible]orks Association, Water Pollution Control Federation, 1975.

[4] "AWWA Standard for Asbestos – Cement Pipe, 4 in. through 24 in. for Water and Other Liquids," AWWA C400-77, Revision of C400-75, AWWA, Denver, Colorado.

[5] "Standard Methods for the Examination of Water and Wastewater," 15th Edition, American Public Health Association, American Water Works Association, Water Pollution Control Federation, 1980.

skills through the successful participation in the analysis of applicable performance evaluation samples.

1.4 Analyst for Organic Contaminants. The following are recommended minimum standards for the analyst position.

1.4.1 Academic training: Minimum of bachelor's degree in chemistry or its equivalent (State certification or licensing may be considered).

1.4.2 Experience: Minimum of 6 months of experience in measurements being considered for certification and 2 years of experience in organic analysis. Each year of college level training in related scientific fields or demonstrated equivalency shall be considered equal to 1 year of work experience. Such a substitution should not exceed one-half of the required experience.

1.4.3 Supervision: Supervision by an analyst (also eligible to analyze for organic chemicals) who has:

1.4.3.1 Minimum of bachelor's degree or its equivalent, with 1 year of course work in organic chemistry.

1.4.3.2 One year of experience in measurement of organic chemicals by gas chromatography.

1.5 GC/MS Operator. In addition to the organic analyst requirements above, the following are recommended minimum standards for the GC/MS operator, if this technique is used.

1.5.1 Training: Satisfactory completion of a minimum one week course in GC/MS offered by equipment manufacturer, professional organization, university, or other qualified operator.

1.5.2 Experience: Minimum of 1 year experience in the operation of a GC/MS instrument.

2. Laboratory Facilities

The laboratory facilities should be clean, air conditioned and with adequate lighting at the bench top. It is recommended that 150 to 200 square feet/person be available. The laboratory should contain at least 15 linear feet of usable bench space per analyst. The laboratory should have provisions for the disposal of chemical wastes. While safety is not an aspect of laboratory certification, exhaust hoods are recommended for the analysis of trace elements and organics. This includes venting for preparation, extraction and analysis.

3. Laboratory Equipment and Supplies

The specifications for instruments that are required for the measurement of chemistry contaminants can be found under Critical Elements for Certification for Chemistry. In addition, it is recommended that a laboratory purchase equipment that meets the specifications below.

3.1 Muffle furnace: Capable of heating glassware to 400°C for cleaning.

3.2 Centrifuge: Capable of handling, as a minimum, 15 mL centrifuge tubes.

3.3 Refrigerator: For storing organics and flammable materials, an "explosion-proof" type of refrigerator should be used. When refrigeration is not required, an explosion-proof cabinet may be used.

3.4 Glassware: Should be of borosilicate glass, which is more resistant than regular soft glass to damage by heat, chemicals, and abuse. All volumetric glassware should be marked Class A, denoting that it meets Federal Specifications and need not be calibrated before use.

3.5 GC/MS interface: It is recommended that the interface between the end of the chromatographic column and the ion source of the mass spectrometer be constructed with deactivated glass or glass-lined materials. However, the GC/MS interface can use any separator, transfer line, or other interface part, provided it is demonstrated that the system meets the BFB performance specifications.

3.6 GC/MS data system: It is desirable, but not required, that the GC/MS data system have the following additional features:

3.6.1 Ability to perform automatic quantitative analysis using integrated specific ion abundances and either a single internal or external standard.

3.6.2 Ability to perform automatic quantitative analysis using integrated specific ion abundances and regression analysis with multiple internal or external standards.

4. General Laboratory Practices

4.1 General

4.1.1 Chemicals/reagents: "Analytical reagent grade" (AR) chemicals should be used for most analyses required of water treatment laboratories. Consult "Standard Methods for the Examination of Water and Wastewater," 14th edition, part 102, pages 5-8, or the latest edition of this reference, for more detailed information on reagent grades. Individual analytical procedures in "Standard Methods for the Examination of Water and Wastewater," and the U. S. EPA's "Methods for Chemical Analysis of Water and Wastes" may specify special requirements for the reagents to be used.

4.1.2 Laboratory safety: While safety is not an aspect of laboratory certification, evaluators should point out, on an informal basis, potential safety problems observed during an on-site visit.

4.2 Inorganic contaminants

4.2.1 Glassware preparation: All glassware should be washed in a warm detergent solution and thoroughly rinsed first in tap water and then in distilled water. This cleaning procedure is sufficient for most analytical needs, but the individual procedures should be referred to for more elaborate precautions to be taken against contamination of glassware. It has been found advantageous to maintain a separate set of glassware (suitably prepared) for the nitrate, mercury, and lead procedures due to the potentiality for contamination from the laboratory environment.

4.2.2 Distilled/deionized water: Water having resistivity values of 0.5 megohms (2.0 micromhos)/cm at 25°C is satisfactory. Megohms are related to micromhos in the following manner:

$$\frac{1}{\text{megohms}} = \text{micromhos},$$

$$\frac{1}{\text{micromhos}} = \text{megohms}$$

Excellent quality water has resistivity values exceeding 1.0 megohms/cm (less than 1.0 micromhos/cm) at 25°C. High quality water meeting such specifications may be purchased from commercial suppliers; laboratories should request a list of quality specifications for any water purchased. Quality of distilled/deionized water is best maintained by sealing from the atmosphere. Quality checks should be made at planned intervals and documented.

4.3 Organic contaminants

4.3.1 Glassware preparation: All glassware should be washed in a warm detergent solution and thoroughly rinsed first in tap water and then in distilled water. All glassware should have a final rinse with nanograde acetone or its equivalent and should then be air dried in an area free of organic contamination.

4.3.2 Reagent water: Reagent water for organic analysis should be free of interferences that coelute from the gas chromatograph with the compound being measured. It may be necessary to treat distilled water with activated carbon to eliminate all interferences.

5. Analytical Methodology

A list of the approved methodology for inorganic contaminants can be found in Table IV-2. The approved methodology for organic contaminants is listed in Table IV-3.

6. Sample Collection, Handling, and Preservation

The type of sample containers required, preservation techniques, and maximum holding times for all inorganic contaminants can be found in Table IV-4. Table IV-5 identifies these critical elements for the organic contaminants.

7. Quality Assurance

The minimum requirements for quality control are described in Critical Elements for Certification. Performance and documentation of the following quality control practices are strongly recommended.

7.1 Current service contract should be in effect on all balances.

7.2 Class S weights should be available to make periodic checks on balances.

7.3 Thermometer certified by the National Bureau of Standards (or one of equivalent accuracy) should be available to check thermometers in ovens, etc.

7.4 Color standards or their equivalent should be available to verify wavelength settings on spectrophotometers.

7.5 Chemicals should be dated upon receipt of shipment and replaced as needed or before shelf life has been exceeded.

7.6 Additional recommended practices have been established for a laboratory analyzing supply samples other than its own:

7.6.1 Laboratory should analyze a certified laboratory control standard (U.S. EPA Quality Control Sample, or equivalent) once per quarter for the parameters measured. The measured value should be within the control limits established by EPA for each analysis for which the laboratory wishes to be certified.

7.6.2 At least one duplicate sample should be run every 10 samples, or with each set of samples, to verify precision of the method. Checks should be within the control limits established by EPA for each analysis for which the laboratory wishes to be certified.

7.6.3 Standard deviations should be calculated and documented for all methods being conducted.

7.6.4 Quality control charts or a tabulation of mean and standard deviation or equivalent should be used to document validity of data on an as-run basis.

8. Records and Data Reporting

9. Action Response to Laboratory Results

10. Free Chlorine Residual, Turbidity, pH and Temperature

Free chlorine residual, turbidity, pH and temperature measurements do not need to be done in approved laboratories, but may be performed by any persons acceptable to the State. There is, however, a definite need for quality control guidelines to be instituted at the State level for these measurements; it is equally important that systems be in use to assure validity of data for these critical measurements.

10.1 Methodology – Only approved methodology may be used for free chlorine residual, turbidity, pH and temperature. The approved methods are listed in Table IV-6. All other procedures are considered alternative analytical techniques.

10.2 Sealed liquid turbidity standards purchased from the instrument manufacturer must be calibrated against properly prepared and diluted formazin or styrene divinylbenzene polymer standards at least every 4 months in order to monitor for any eventual deterioration. These standards should be replaced when any major change from the original calibration occurs. Solid turbidity standards composed of plastic, glass, or other materials are not reliable and should not be used.

10.3 Calibration intervals for color wheels, sealed ampules, and other visual standards for free chlorine residuals: Laboratories utilizing visual comparison devices should calibrate the standards incorporated into such devices at least every six months. These calibrations should be documented. Directions for preparing temporary and permanent type visual standards can be found in Method 409F, "Standard Methods for the Examination of Water and Wastewater," 14th edition, published in 1975 by the American Public Health Association. By comparing standards and plotting such a comparison on graph paper, a correction factor can be derived and applied to all future results obtained on the now calibrated apparatus.

10.4 Additional criteria – The following criteria are recommended for use by the State for approval of persons for performing free chlorine residual, turbidity, pH and temperature measurements.

10.4.1 **Free chlorine residual** – Samples may be collected in plastic or glass. Samples are not preserved; analyses are to be made as soon as practicable, or within 1 hour. A DPD Colorimetric Test Kit, spectrophotometer, or photometer is required.

10.4.2 **Turbidity** – Samples may be collected in plastic or glass. Samples are not preserved; analyses are to be made as soon as practicable, or within 1 hour. Nephelometer is needed with light source for illuminating the sample and one or more photoelectric detectors with a readout device to indicate the intensity of light scattered at right angles to the path of the incident light. Unit may be line/bench or battery/portable operated.

10.4.3 **pH** – Samples may be collected in plastic or glass. Samples are not preserved; analyses are to be made as soon as practicable, or within 1 hour. A pH meter is necessary.

10.4.4 **Temperature** – Samples are to be analyzed immediately. Requires any good grade mercury-filled or dial type centigrade thermometer, or a thermistor.

Sample Forms for On-Site Evaluation of Laboratories Involved in Analysis of Public Water Supplies—Chemistry

Laboratory: _____

Street: _____

City: _____ State: _____

Survey By: _____

Affiliation: _____

Date: _____ Telephone No.: _____

Laboratory _____ Evaluator _____

Location _____ Date _____

Personnel

Position/Title	Name	Academic Training			Present Specialty	Experience (years and area)
		HS	BA/BS	Other		
Laboratory Director						
Supervisor						
Inorganic analysts						
Organic analysts						
GC/MS operators						

Laboratory _____ Evaluator _____

Location _____ Date _____

General Laboratory Equipment and Instruments

Item	No. of Units	Manufacturer	Model	Satisfactory	
				Yes	No
Analytical balance					
Magnetic stirrer					
pH meter					
Conductivity meter					
Hot plate					
Refrigerator					
Drying oven					
Thermometer					

Laboratory _____ Evaluator _____

Location _____ Date _____

Laboratory Equipment—Inorganic Contaminants

Item	No. of Units	Manufacturer	Model	Satisfactory Yes	No	N/A
Photometer						
Spectrophotometer						
Filter photometer						
Specific ion meter						
Electrodes						
Stirred water bath						
Automated analysis system						
Sampler						
Proportioning pump						
Manifold or cartridge						
Heating bath						
Bath with distilling head						
Continuous filter						
Colorimeter						
ISE detector						
Recorder						
Arsine generator						

Laboratory _____ Evaluator _____

Location _____ Date _____

Laboratory Equipment—Inorganic Contaminants

Item	No. of Units	Manufacturer	Model	Satisfactory Yes	No	N/A
Atomic absorption spectrophotometer						
Readout system						
Fuel and oxidants						
Burner						
Hollow cathode lamps						
Graphite furnace						
Pipets						
Background corrector						
Separatory funnels						
Hydride generation system						
Mercury analyzer						
Absorption cell						
Air pump						
Flow meter						
Spectrophotometer						
Aeration tube						
Drying unit						

Laboratory _____ Evaluator _____

Location _____ Date _____

Laboratory Equipment—Organic Contaminants

Item	No. of Units	Manufacturer	Model	Satisfactory		
				Yes	No	N/A
Gas chromatography						
Chlorinated hydrocarbons						
Chlorophenoxys						
TTHM by purge and trap						
TTHM by solvent extraction						
TTHM by GC/MS						
Recorder						
Purge and trap system						
Purging device						
Trapping device						
Desorber assembly						
Kuderna-Danish sets						
Water bath						

Laboratory _____ Evaluator _____

Location _____ Date _____

Methodology

Contaminant	Name or Description of Method	Reference (Cite Source and Method by Number or Page and Year)	Sample Load per Month	Satisfactory	
				Yes	No
Inorganic					
Arsenic					
Barium					
Cadmium					
Chromium					
Fluoride					
Lead					
Mercury					
Nitrate					
Selenium					
Silver					
Organic					
Chlorinated hydrocarbons					
Chlorophenoxys					
TTHM					
MTP					

Laboratory _____ Evaluator _____

Location _____ Date _____

Sample Handing and Preservation

Contaminant	Container Used (Material and Size)	Preservative Used	Maximum Holding Time	Satisfactory Yes	Satisfactory No
Inorganic					
Arsenic					
Barium					
Cadmium					
Chromium					
Fluoride					
Lead					
Mercury					
Nitrate					
Selenium					
Silver					
Organic					
Chlorinated hydrocarbons					
Chlorophenoxys					
TTHM					
MTP					

Laboratory _____ Evaluator _____

Location _____ Date _____

Sample Collection

Item	Comments	Satisfactory Yes	Satisfactory No
General			
Trained sample collector			
Representative sampling			
Complete sample form			
Inorganic			
Appropriate sampling and preservation			
Overaged samples discarded			
Organic			
Appropriate sampling and preservation			
TTHM stabilizer added in laboratory			
TTHM hermetic seal			
Overaged samples discarded			

Laboratory _____ Evaluator _____

Location _____ Date _____

Quality Assurance and Data Reporting

Item	Comments	Satisfactory Yes	Satisfactory No
General			
QA plan and data			
Annual performance samples analyzed			
Methods manual available			
Records kept 3 years			
pH meter calibration			
Inorganic			
Standard curve			
Daily calibration check			
Check after 20 samples			
Organic			
Daily method blank			
Daily calibration			
Quarterly QC samples			
TTHM field blanks			
10% TTHM in duplicate			
TTHM control standards			
TTHM startup test			
501.2 Source water blank check			
BFB tuning check			

Chapter V

Microbiology

Critical Elements for Certification

The technical criteria in this chapter are divided into two sections: Critical Elements for Certification and Recommended Practices. Critical Elements for Certification are necessary for generation of valid data and certification, and consequently will be used to determine certification status. All quality control items are designated as "QC". "QC" items in the Critical Elements for Certification section necessitate written records.

1. Personnel

Although there are no certification requirements for laboratory analysts, laboratory administrators and evaluators should recognize the importance of relevant training and experience to the acquisition of valid compliance monitoring data. Recommended minimum standards can be found under Recommended Practices.

2. Laboratory Facilities

There are no critical elements for laboratory facilities. Recommended guidelines can be found under Recommended Practices.

3. Laboratory Equipment and Supplies

Laboratories must have available or have working access to items listed below for analyzing potable water samples for the detection of coliform organisms.

A laboratory may request or contract another laboratory to conduct specified quality control testing, e.g., testing the quality of laboratory pure water (4.3.2); calibration of non-reference weights (3.2.2); and calibration of temperature monitoring devices (3.3.2). The laboratory conducting the actual quality control test(s) must be certified for the specific parameter and provide copies of quality control data to the requesting laboratory. Therefore, the requesting laboratory is not required to have equipment, supplies, and materials to conduct specified quality control tests.

3.1 pH Meter

3.1.1 Accuracy and scale graduations within ± 0.1 units.

3.1.2 Use pH buffer aliquot only once.

QC 3.1.3 Standardize pH meter each use period with pH 7.0 standard buffer.

3.2 Balance (top loader or pan)

3.2.1 Balance detects 100 mg at a 150 gram load.

QC 3.2.2 Calibrate balance monthly, using Class S or S-1 reference weights or weights traceable to Class S or S-1 weights. If non-reference weights are used, calibrate annually with Class S or S-1 reference weights.

QC 3.2.3 Maintain service contract or internal maintenance protocol and maintenance records.

3.3 Temperature Monitoring Device

3.3.1 Glass/mercury or dial thermometers used in incubator units graduated in 0.5°C increments. Mercury column for glass thermometers is not separated.

QC 3.3.2 Check calibration of glass/mercury thermometers annually and dial thermometers quarterly against a reference NBS thermometer or one which meets the requirements of NBS monograph 150.

3.4 Incubation Unit

3.4.1 The incubator unit has an internal temperature monitoring device and maintains a temperature of 35 ± 0.5°C. For nonportable incubators, thermometers are placed on the top and bottom shelves of the use area, the thermometer bulb immersed in liquid. If an aluminum block is used, culture dishes and tubes fit snugly.

QC 3.4.2 Record temperature for days in use at least twice per day with each reading separated by at least 4 hours.

3.5 Autoclave

3.5.1 The autoclave has a temperature gauge with a sensor on the exhaust, and an operational safety valve. Maintains sterilization temperature during the sterilizing cycle and completes an entire cycle within 45 minutes when a 12-15 minute sterilization period is used. Autoclave depressurizes slowly to insure media do not boil over and bubbles do not form in inverted tubes.

3.5.2 Because of safety concerns and difficulties with operational control, approval of pressure cookers and vertical autoclaves requires QC data demonstrating sterility and proper media reactions.

QC 3.5.3 Record date and sterilization time and temperature for each cycle. Establish service contract or internal maintenance protocol, and maintain records.

3.6 Conductivity Meter (needed only for laboratories analyzing laboratory pure water; optional for laboratories which send water to another certified laboratory for analysis).

3.6.1 Conductivity meter graduated in ohms or mhos with a sensitivity of at least 0.33%. Has a range from 2 ohms to 2 megohms or equivalent micromhos ± 1%.

3.7 Refrigerator

3.7.1 Refrigerator maintains a temperature of 1°C to 5°C. Thermometer graduated in at least 1°C increments and the thermometer bulb immersed in liquid.

QC 3.7.2 Record temperatures for days in use at least once per day.

3.8 Inoculating Equipment

3.8.1 Metal or plastic loops, or dry heat-sterilized hardwood applicator sticks.

3.9 Membrane Filtration Equipment (needed if MF procedure is used).

3.9.1 MF units are stainless steel, glass, or autoclavable plastic. Not scratched, corroded, and do not leak.

3.9.2 10 to 15X magnification device with fluorescent light source used to count sheen colonies.

3.9.3 Membrane filters approved by the manufacturer for total coliform water analysis. Approval based on data relating to toxicity, recovery, retention, and absence of growth-promoting substances. Filters are cellulose ester, white, gridmarked, 47 mm diameter, and 0.45 um pore size. Alternate pore sizes may be used if manufacturer provides performance data equal to or better than the 0.45 um pore size. Membrane filters are presterilized or autoclaved before use.

3.10 Culture Dishes (Loose or Tight Lid)

3.10.1 Presterilized plastic or sterilizable glass culture dishes are used. To maintain sterility of glass culture dishes, use stainless steel or aluminum canisters, or wrap dishes in a heavy aluminum foil or char-resistant paper.

3.10.2 Loose-lid dishes are incubated in a tight-fitting container, e.g., plastic vegetable crisper, to prevent dehydration of membrane filter and medium.

3.10.3 Opened packs of disposable culture dishes resealed between major uses.

3.11 Pipets

3.11.1 To sterilize and maintain sterility of glass pipets, use stainless steel or aluminum canisters, or wrap individual pipets in char-resistant paper.

3.11.2 Opened packs of disposable sterile pipets are resealed between major use periods.

3.12 Culture Tubes and Closures

3.12.1 Tubes are made of borosilicate glass or other corrosion-resistant glass.

3.12.2 Culture tubes used for Presumptive Test in Fermentation Tube procedure (MPN) are of a sufficient size to contain medium plus sample without being more than 3/4 full.

3.12.3 Tube closures are stainless steel, plastic, aluminum, or loosened screw caps with non-toxic liners. Cotton plugs are not acceptable.

3.13 Sample Containers

3.13.1 Sample bottles are wide-mouth plastic or non-corrosive glass with either screw cap with non-toxic liner or ground glass stopper, which withstand repeated sterilization, or other USEPA-approved sample containers. Capacity is at least 120 mL (4 oz.).

3.13.2 Glass-stoppered bottle closures are covered with aluminum foil or char-resistant paper for sterilization.

3.14 Glassware and Plasticware

3.14.1 Glassware is made of borosilicate glass or other corrosion-resistant glass and free of chips and cracks. Markings on graduated cylinders and pipets are legible. Plastic items are clear and nontoxic.

3.14.2 Graduated cylinders for measurement of sample volumes have a tolerance of 2.5% or better.

3.14.3 Pipets delivering volumes of 10 mL or less are accurate within a 2.5% tolerance.

4. General Laboratory Practices

4.1 Autoclave Sterilization Procedures

4.1.1 The times for autoclaving materials at 121°C are listed. Except for membrane filters and pads and carbohydrate-containing media, indicated times are minimal times which may necessitate adjustment depending upon volumes, containers and loads.

Item	Time (minutes)
Membrane filters & pads	10
Carbohydrate containing media	12-15
Contaminated test materials	30
Membrane filter assemblies	15
Sample collection bottles	15
Individual glassware	15
Dilution water blank	15
Rinse water	15

4.1.2 Autoclaved membrane filters and pads and all media are removed immediately after completion of cycle.

4.1.3 Metal filtration equipment is autoclaved between filtration series. Glass and plastic filtration equipment is sterilized between filtration series. A filtration series ends when 30 minutes or longer elapse between sample filtrations.

4.2 Sample Containers

4.2.1 Sodium thiosulfate (100 mg/L) should be added before sterilization to sample containers (0.1 mL of 10% sodium thiosulfate solution per 120 mL container).

QC 4.2.2 Select at least one bottle at random from each batch and confirm sterility by adding approximately a 25-mL volume of a sterile non-selective broth. Incubate at 35° ± 0.5°C for 24 hours and check for growth.

4.3 Laboratory Pure Water

4.3.1 Only satisfactorily-tested laboratory pure water from stills or deionization units is used to prepare media, reagents, and dilution/rinse water for performing bacteriological analyses.

QC 4.3.2 Test the quality of the laboratory pure water or have it tested by another certified laboratory to assure it meets the following requirements:

Parameter	Requirements	Frequency
Conductivity	>0.5 megohms resistance or <2 micromhos/cm at 25°C	Monthly
Total Chlorine Residual	Non-detectable	Monthly
Quality of Laboratory Pure Water*	Ratio 0.8-3.0	Annually

*Test for bacteriological quality of distilled water (**Standard Methods**, p. 888; also **Microbiological Methods for Monitoring the Environment** EPA-600/8-78-017, p. 200).

4.4 Dilution/Rinse Water

4.4.1 Stock buffer solution is prepared according to **Standard Methods** p. 892, or **Microbiological Methods for Monitoring the Environment**, p. 57, using laboratory pure water.

4.4.2 Stock buffer is autoclaved or filter-sterilized, labeled and dated. Insure stored stock buffer is free of turbidity.

4.4.3 Dilution/rinse water is prepared by adding 1.25 mL of stock buffer solution and 5 mL of $MgCl_2$ solution (38g $MgCl_2$/L) per liter of laboratory pure water and sterilize. Do not use $MgSO_4 \cdot 7H_2O$ because of possible precipitation problems. The pH of

References for Chapter V: **Standard Methods for the Examination of Water and Wastewater**, 14th ed.; **Microbiological Methods for Monitoring the Environment** EPA-600/8-78-017.

both the stock buffer solution and the final dilution/rinse water is 7.2 ± 0.2.

QC 4.4.4 Check each batch of rinse water for sterility by adding 20 mL of water to a 100-mL volume of a non-selective broth. Incubate at 35°C ± 0.5°C for 24 hours and check for growth.

4.5 Glassware Washing

4.5.1 Distilled or deionized water is used for final rinse.

QC 4.5.2 Perform the Inhibitory Residue Test (**Standard Methods,** p. 885, and **Microbiological Methods for Monitoring the Environment,** p. 199) on initial use of a washing compound and whenever a different formulation of washing compound is used in order to insure that glassware is free of toxic residue.

4.6 Media—General Requirements

4.6.1 Dehydrated or pre-prepared media manufactured commercially is used. Store media in cool, dry location and discard caked or discolored dehydrated media.

QC 4.6.2 For media prepared, record the date of preparation, type of medium, lot number, sterilization time and temperature, final pH, technician's initials.

4.7 Membrane Filter (MF) Media (needed only if laboratory conducts MF procedure)

4.7.1 m-Endo broth or agar in the single step procedure, or m-Endo agar LES in the enrichment technique is used. Insure that ethanol used in rehydration procedure is not denatured. To dissolve medium, use boiling water bath (or direct heat if constantly attended) to bring medium just to boiling point. Do not boil medium. Final pH 7.2 ± 0.2 C.

4.7.2 MF broth, MF agar medium, and ampouled m-Endo broth are refrigerated no longer than 96 hours, 2 weeks, and manufacturer's expiration date, respectively.

4.8 Most Probable Number (MPN) Media

4.8.1 Double strength lauryl tryptose (lauryl sulfate) broth or lactose broth is used for Presumptive Test and single strength brilliant green lactose bile broth for Confirmed Test. Dispense broth medium volume of not less than 10 mL per tube and autoclave media at 121°C for 12-15 minutes. Final pH 6.8 ± 0.2 (7.2 ± 0.2 for brilliant green broth).

4.8.2 If MPN media are refrigerated after sterilization, incubate overnight at 35°C before use. Discard tubes showing growth and/or bubbles. Use MPN media prepared in tubes with loose-fitting closures within one week. Store broth media in screw cap tubes no longer than 3 months, provided media are stored in dark. Discard media if evaporation exceeds 10% of original volume.

4.8.3 If Levine Eosin Methylene blue (EMB) agar is used for Completed Test, dissolve by using boiling water bath (or direct heat if constantly attended) to bring medium just to boiling point. Autoclave medium at 121°C for 12-15 minutes. Final pH 7.1 ± 0.2. Use medium not autoclaved on the day of preparation; DO NOT STORE. Autoclaved agar which is refrigerated is used within two weeks.

4.9 Standard Plate Count Medium (SPC)

4.9.1 SPC agar is autoclaved at 121°C for 15 minutes, depending upon volume. Final pH 7.0 ± 0.2. Temper melted agar between 44°C to 46°C before pouring. Hold melted agar no longer than 8 hours. Do not melt sterile medium more than once.

5. Analytical Methodology

5.1 Approved analytical methodology is stated in the National Interim Primary Drinking Water Regulations (NIPDWR), as amended. Do not use tentative methods, equipment, or procedures unless they satisfy the requirements of Section 141.27 of the NIPDWR.

5.2 MF Procedure

5.2.1 Sample volumes analyzed by the MF procedure must be 100 mL ± 2.5 mL.

5.2.2 Confluent growth is defined as bacterial growth with or without sheen covering the entire membrane filter.

TNTC (too numerous to count) is defined as greater than 200 total bacterial colonies on the membrane filter.

5.2.3 Samples resulting in confluent growth or TNTC with less than five distinguishable sheen colonies are invalid. Record as "confluent growth" or "TNTC" and request a new sample from the same sampling site.

5.2.4 Samples resulting in confluent growth or TNTC with five or more distinguishable sheen colonies may be a MCL violation. Report as "confluent growth" or "TNTC" with the number of distinguishable sheen colonies. Check samples must be obtained from the same sampling site.

5.2.5 Verify all sheen colonies regardless of the amount of sheen when the number of the sheen colonies is five to and including 10/100 mL. When the number of sheen colonies exceeds 10/100 mL, randomly pick and verify at least 10 colonies representative of all sheen types.

5.2.6 Adjust initial counts based only upon verification data.

QC 5.2.7 Conduct MF sterility check (rinse water, medium, and supplies) at the end of each filtration series. If controls indicate contamination, reject all data on samples affected and request immediate resampling of the samples involved.

5.3 MPN Procedure

5.3.1 Conduct MPN Completed Test on all unsatisfactory samples (> three positive tubes). Gram-staining is optional for potable water samples.

5.3.2 For unsatisfactory samples, adjust the number of positive tubes on the basis of the Completed Test.

5.3.3 If the MPN test is used on water supplies that have a history of confluent growth or TNTC by the MF procedure, all presumptive tubes with heavy growth without gas production are submitted to the Confirmed Test to check for coliform suppression.

QC 5.3.4 If no positive tubes result from potable water samples, perform the MPN procedure, including the Completed Test, quarterly on all positive confirmed tubes from at least one coliform-positive water sample.

6. Sample Collection, Handling, and Preservation

(applicable to those laboratories delegated responsibility for sample collection. All laboratories are responsible for items 6.4 and 6.5).

6.1 Sampling frequency must conform to that specified by the regulations. Collector is trained in sampling procedures and approved by the appropriate regulatory authority or its designated representative.

6.2 Samples must be representative of the potable water distribution system.

Water taps used for sampling are free of aerators, strainers, hose attachments, mixing type faucets and purification devices. A steady water flow should be maintained for at least 2 minutes to clear the service line before sampling. Collect at least a 100 mL sample, allowing ample air space to facilitate mixing of sampling by shaking. Immediately after collection, enter on the sample report form the sample site location, sample type (e.g., routine, check), date and time of collection, free chlorine residual, collector's initials, and any remarks.

6.3 Applicable State regulations pertaining to chain-of-custody are followed.

6.4 The report form includes the date and time of sample arrival at the laboratory and the date and time analysis begins. Record additional information as required by the NIPDWR, Section 141.33.

6.5 Holding/transit time between sampling and analysis does not exceed 30 hours. If the laboratory is required by State regulation to examine samples after 30 hours and up to 48 hours, the laboratory is to indicate that the data may be invalid because of excessive delay before sample processing. Samples arriving after 48 hours shall be refused without exception and a new sample requested.

7. Quality Assurance

7.1 The laboratory prepares and follows a written QA plan (see Chapter III, section on QA plans).

7.2 All items under Critical Elements for Certification which are designated as quality control by the "QC" notation relating to analytical quality control and quality control checks on media, equipment, and materials, necessitate written records which are retained by the laboratory for 5 years.

QC **7.3** A quality control sample is analyzed annually (when available).

QC **7.4** An unknown performance evaluation sample is satisfactorily analyzed annually (when available).

8. Records and Data Reporting

Records of microbiological analyses must be kept by the laboratory or must be accessible to the laboratory for at least 5 years. Actual laboratory reports may be kept, or data may be transferred to tabular summaries, provided that the following information is included:

8.1 Date, place, and time of sampling, name of persons who collected the sample.

8.2 Identification of sample as to whether it is a routine distribution system sample, check sample, raw or process water sample, or other special purpose sample.

8.3 Date of receipt of sample and analysis.

8.4 Laboratory and persons responsible for performing analysis.

8.5 Analytical technique/method used.

8.6 Results of analysis.

9. Action Response to Laboratory Results

Promptly notify the proper authorities of unsatisfactory sample results on the basis of MPN Confirmed Test or unverified MF coliform data, and check sampling from the same sampling point must be initiated.

Recommended Practices

All laboratories should perform a minimum of 20 coliform examinations monthly, preferably five per week, by either the MPN or MF procedures to qualify for certification and to maintain certification status. This will insure that the analyst maintains expertise in water bacteriological methodology.

The minimum number of coliform examinations (20) may be performed on a variety of water sample types collected from different stages of the water treatment process, raw source water, surface or ground water, as well as potable water samples collected from a distribution system.

1. Personnel

1.1 Analyst

The analyst performs microbiological tests with minimal supervision.

Academic Training: Minimum of high school education.

Job Training: One week of training in microbiological analysis of drinking water acceptable to the State (or U.S. EPA for non-primacy States), plus a minimum of 30 days on-the-job training. Personnel should take advantage of sources available from Federal and State regulatory agencies.

1.2 Supervisor/Consultant

Supervision by a professional scientist experienced in water microbiology. If a supervisor is not available, a consultant having the same qualifications may be substituted. State laboratory personnel would be a primary source for consultations.

Academic Training: Minimum of a bachelor's degree in microbiology, biology, chemistry, or a closely related field.

Job Training: Minimum of two weeks training from a Federal agency, State agency, or academic institution in microbiological analysis of drinking water.

Experience: At least one year of bench experience in sanitary (water, milk, or food) microbiology.

2. Laboratory Facilities

Laboratory space should be adequate (200 ft^2 and 6 linear ft of bench per analyst) to accommodate peak workload. Work space should include sufficient bench-top area for processing samples; storage space for media, glassware, and portable equipment; floor space for stationary equipment (incubators, waterbaths, refrigerators, etc.); and associated area for cleaning glassware and for sterilizing materials. In small water plant laboratories, the space required for laboratory analyses and preparation may be consolidated into one room with the various functions allocated to different parts of the room. Facilities should be clean, air-condi-

tioned, and with adequate lighting at the bench top (100 ft. candles).

Laboratory safety should involve a conscious effort to safeguard against electrical shock, fire, accidental chemical spills, and to minimize microbiological hazards, facility deficiencies, and equipment failures. While safety is not an aspect of laboratory certification, the evaluator should informally indicate potential safety problems observed during an on-site visit.

3. Laboratory Equipment and Supplies

3.1 pH meter

3.1.1 Electrodes are maintained according to manufacturer recommendations.

QC 3.1.2 Date commercial buffer solution container upon receipt and discard before expiration date.

QC 3.1.3 Check for accuracy quarterly, using NBS standard reference materials.

3.2 Temperature Monitoring Device

3.2.1 The calibration of any continuous recording device used to monitor incubator temperature should be checked annually by determining whether it is sensitive to within 1°C of a reference NBS thermometer.

3.3 Incubator Unit

3.3.1 A continuous recording device should be used to monitor general operation of incubator unit.

3.4 Autoclave

3.4.1 In addition to temperature gauge, autoclave should have pressure gauge. Avoid overcrowding in autoclave, and monitor temperature throughout sterilization cycle.

QC 3.4.2 Use heat sensitive tapes, spore strips or ampoules, or maximum-temperature-registering thermometer during autoclave cycle.

QC 3.4.3 Check automatic timing mechanism with stopwatch.

3.5 Hot Air Oven

3.5.1 The oven should maintain a stable sterilization temperature (170°C-180°C). Sterilize only dried items, avoid overcrowding, and use heat sensitive tape to insure sterilization time is attained. The oven thermometer should be graduated in at least 10°C increments, with the bulb placed in sand during use.

QC 3.5.2 Record date, sterilization time and temperature of each cycle.

3.6 Colony Counter

3.6.1 A colony counter, dark field model, should be used to count standard plate count colonies.

3.6.2 A mechanical hand tally for counting bacterial colonies should be used.

3.7 Membrane Filters and Pads

QC 3.7.1 Record lot number and date received.

QC 3.7.2 Test recovery and performance of membranes compared to a previously acceptable lot.

4. General Laboratory Practices

4.1 Sterilization Procedures

4.1.1 As an alternative to autoclaving, dried glassware can be sterilized in an oven (170°-180°C for a minimum of 2 hours).

4.1.2 The membrane filter assembly should be exposed to UV light or boiling water for at least 2 minutes to prevent bacterial carryover between filtrations.

4.2 Laboratory Pure Water

QC 4.2.1 Test the bacteriological quality of the laboratory pure water monthly by the Use Test (EPA **Microbiological Methods for Monitoring the Environment,** p. 203).

QC 4.2.2 If laboratory pure water does not meet quality requirements, perform the following supplementary to determine cause:

Parameter	Limits
Pb, Cd, Cr, Cu, Ni, Zn	Not greater than 0.5 mg/L
Total metals not limited to those above	Equal to or less than 1.0 mg/L
pH	5.5 - 7.5
Standard Plate Count	< 1,000/mL

4.3 Dilution Water

QC 4.3.1 Perform sterility check by adding 20 mL of water to 100 mL volume of non-selective broth, incubate at 35° ± 0.5°C for 24 hours, and check for growth. Discard if contaminated.

4.4 Media

4.4.1 Media should be ordered on a 12-month need basis. Purchase 1/4 lb. bottles, except for large volume uses, and date bottles upon receipt and also when initially opened. Determine recovery and performance of new media compared to previously acceptable lots by using positive and negative control cultures.

4.4.2 Dehydrated media should be discarded 6 months after opening; if stored in a desiccator, storage may be extended to 12 months. Discard unopened dehydrated media after 2 years.

4.4.3 Sterile flasks should be used for preparation of MF medium.

5. Analytical Methodology

5.1 The analyst has the option of conducting either the MPN or MF procedure; however, the MF procedure is preferred when applicable because it permits analysis of a larger sample volume in reduced analytical time.

QC 5.2 Laboratories which conduct the MF procedure and have two or more analysts should analyze one known coliform-positive sample monthly and each analyst should count the sheen colonies on the same membrane. The counts should agree within 10%. This insures that the analysts are capable of distinguishing sheen from non-sheen colonies.

QC 5.3 At least one non-sheen colony of each morphological type should be verified monthly.

6. Sample Collection, Handling, and Preservation

6.1 Samples should be iced during transit, and refrigerated at 1°C-5°C during storage in the laboratory.

6.2 Samples delivered by collectors to the laboratory should be analyzed on the day of collection. (This item may become a Critical Element for Certification).

7. Quality Assurance

8. Records and Data Reporting

Proper authorities should be alerted to the occurrence of high background levels of noncoliform organisms by the MF procedure; or turbid tubes lacking gas, using the MPN procedure; or heterotrophic bacterial densities exceeding 500 colonies/mL.

9. Action Response to Laboratory Results

Although check sampling is to be initiated on the basis of MPN Confirmed Test and unverified MF coliform counts, data used to determine monthly compliance may be adjusted by conducting the Completed Test for the MPN procedure and/or verifying MF counts.

Sample Forms for On-Site Evaluation of Laboratories Involved in Analysis of Public Water Supplies—Microbiology

Laboratory _____ _____

Street _____ _____

City _____ _____ State _____

Telephone Number _____

Survey By _____

Affiliation _____

Date _____

Codes for Marking On-Site Evaluation Forms S - Satisfactory X - Unsatisfactory U - Undetermined NA - Not Applicable

1. Personnel

Position/title	Name	Academic training HS	BA/BS	MA/MS	Ph.D.	Present specialty	Experience (years/area)
Laboratory director							
Supervisor/ consultant							
Professionals (note discipline)							
Technician/ analyst							

Laboratory _____ Evaluator _____

Location _____ Date _____

2. Laboratory Facilities is Omitted

3. Laboratory Equipment, Supplies, and Materials

3.1 pH Meter

Manufacturer _____ Model _____

Accuracy, ± 0.1 unit _____
Scale graduation, 0.1 units _____
Use pH buffer aliquot only once _____
QC Standardize pH meter each use period with pH 7.0 standard buffer _____

3.2 Balance (Top Loader or Pan)

Manufacturer _____ Model _____

Detects 100 mg at a 150 gram load _____
QC Calibrate balance performance using Class S or S-1 reference weights. If non-reference weights used, they are calibrated initially with Class S or S-1 reference weights. _____
QC Service contract or internal maintenance protocol and record maintained _____

3.3 Temperature Monitoring Device

Glass/mercury or dial thermometer used in incubator units graduated in 0.5°C increments _____
No separation in mercury column _____
QC Check calibration of glass/mercury thermometer annually and dial thermometer quarterly against a reference NBS thermometer or one meeting NBS monograph 150 requirements _____

3.4 Incubation Unit

Manufacturer _____ Model _____

Maintains internal temperature of 35° ± 0.5°C _____
Thermometers placed on top and bottom shelves in use area of non-portable incubators _____
Immerse thermometer bulb in liquid _____
Culture dishes and tubes fit snugly in aluminum block incubator _____
QC Record temperature morning and afternoon for days in use _____

3.5 Autoclave

Pressure cooker/vertical autoclave _____

Manufacturer _____ Model _____

Temperature gauge with sensor on exhaust _____
Operational safety valve _____
Maintains sterilization temperature during cycle _____
Completes entire cycle within 45 minutes when a 12-15 minute sterilization period is used _____
Depressurizes sufficiently slowly to insure media do not boil over and bubbles do not form in fermentation tubes _____
Approval of pressure cookers and vertical autoclaves requires quality control data demonstrating sterility and proper media reactions _____
QC Record date, sterilization time, and temperature for each cycle _____
QC Establish service contract or internal maintenance protocol _____

3.6 Conductivity Meter

Manufacturer _____ Model _____

Graduated in ohms or mhos; range of 2 ohms to 2 megohms or equivalent micromhos ± 1 percent; sensitivity of 0.33 percent or better _____

3.7 Refrigerator

Maintain temperature at 1°C to 5°C
Thermometer graduated in 1° increments
Immerse thermometer bulb in liquid
QC Record temperature for days in use

3.8 Inoculating Equipment

Metal or plastic loops, or dry heat sterilized applicator sticks

3.9 Membrane Filtration Equipment

Manufacturer _____ Type _____

Stainless steel, glass or autoclavable plastic
Units non-leaking, unscratched, not corroded
10 to 15X magnification device with fluorescent light source
Forceps, tips without corrugations

3.10 Membrane Filters and Pads

Manufacturer _____ Type _____

Made from cellulose ester material, white, gridmarked, 47 mm diameter, 0.45 um pore size
Alternate pore size used
Membranes recommended by manufacturer for total coliform water analysis
Membranes and pads are presterilized or autoclaved

3.11 Culture Dishes

Presterilized plastic or sterilized glass dishes used
Loose-lid dishes incubated in a tight-fitting container
Glass culture dishes are sterilized in stainless steel or aluminum canisters or in heavy aluminum foil or char-resistant paper
Open packs of disposable culture dishes are resealed between uses

3.12 Pipets

Glass pipets sterilized in stainless steel or aluminum canisters or individual pipets wrapped in char-resistant paper
Reseal packs of disposable sterile pipets between major use periods
Pipets not etched, mouthpiece and tip are not chipped, graduation markings legible

3.13 Culture Tubes and Closures

Tubes are borosilicate glass or other corrosion-resistant glass
Culture tubes are of sufficient size that medium plus sample does not exceed 3/4 full
Closures are stainless steel, plastic, aluminum, or loosened screw caps with non-toxic liner

3.14 Sample Containers

Capacity at least 120 mL (4 oz.)
Wide-mouth plastic or glass bottle with screw cap or non-corrosive glass bottle with ground glass stopper
Non-toxic liner in screw caps
Glass-stoppered bottle top covered with aluminum foil or char-resistant paper before sterilization

3.15 Glassware and Plasticware

Glass made of borosilicate or other corrosive-resistant glass
Free of chips and cracks
Graduation marks are legible
Plastic items are clear and non-toxic
Graduated cylinders used to measure sample volume have a 2.5% tolerance or better
Pipets used to measure sample volumes have a 2.5% tolerance or better

Laboratory _____ Evaluator _____

Location _____ Date _____

4. General Laboratory Practices

4.1 Autoclave Sterilization Procedures at 121°C

Item	Time	
Membrane filter and pads	10 min	_____
Carbohydrate media	12-15 min	_____
Contaminated test materials	30 min	_____
Membrane filter assemblies	15 min	_____
Sample collection bottles	15 min	_____
Individual glassware	15 min	_____
Dilution water blanks	15 min	_____
Rinse water	15 min	_____

Autoclaved MF filters and pads and all media are removed immediately after sterilization cycle _____

Membrane filter assemblies are sterilized at start of each filtration series _____

4.2 Sample Containers

Sodium thiosulfate added to sample containers before sterilization _____

QC At least one bottle per batch checked for sterility _____

4.3 Laboratory Pure Water

Laboratory pure water is used to prepare media, reagents, and dilution/rinse water _____

QC Requirements for laboratory pure water: _____

Parameters	Frequency	
(a) conductivity of >0.5 megohms or <2 micromhos at 25°C	Monthly	_____
(b) total chlorine residual non-detectable	Monthly	_____
(c) test for bacteriological quality for laboratory pure water, ratio of 0.8-3.0	Annually	_____

4.4 Dilution/Rinse Water

Stock buffer prepared according to Standard Methods and/or EPA Manual 600/8-78-017 _____

Stock buffer autoclaved or filter sterilized, labeled, and dated, and stock buffer free of turbidity _____

Dilution/rinse water is prepared by adding 1.25 mL of stock buffer solution and 5mL of $MgCl_2$ solution per liter of laboratory pure water _____

QC pH of stock buffer solution is 7.2 ± 0.2 _____

QC pH dilution/rinse water 7.2 ± 0.2, adjust pH if necessary _____

QC Rinse water checked for sterility _____

4.5 Glassware Washing

Distilled or deionized water used for final rinse _____

QC Inhibitory residue test performed on clean glassware _____

4.6 Media (General Needs)

Commercially prepared dehydrated media used _____

Dehydrated media stored in cool, dry location _____

Check media pH, adjust if necessary _____

QC Record for media prepared:

(a) date of preparation _____
(b) type of medium _____
(c) lot number _____
(d) sterilization time and temperature _____
(e) final pH _____
(f) technician's initials _____

4.7 Membrane Filter Media

m-Endo broth or agar; final pH 7.2 ± 0.2
m-Endo agar LES; final pH 7.2 ± 0.2

Dissolution of m-Endo broth or agar and m-Endo agar LES:

(a) boiling water bath
(b) direct heat

Use only ethanol
Membrane filter broth refrigerated no longer than 96 hours
Membrane filter agar refrigerated no longer than 2 weeks
Ampouled m-Endo broth refrigerated in accord with manufacturer's expiration date

4.8 MPN Media

Lauryl tryptose (lauryl sulfate) broth
Lactose broth
Broth medium dispensed in volumes not less than 10 mL/tube
MPN media in tubes with loose-fitting closures used within one week
MPN media in screw cap tubes stored no longer than three months; discarded if evaporation exceeds 10% of original volume
Overnight incubation at 35°C of refrigerated sterilized MPN media

Lauryl tryptose (lauryl sulfate) broth

(a) autoclave at 121°C for 12-15 minutes
(b) double strength; final pH 6.7 ± 0.2

Lactose broth

(a) autoclave at 121°C for 12-15 minutes
(b) double strength; final pH 6.7 ± 0.2

Brilliant green lactose bile broth

(a) autoclave at 121°C for 12-15 minutes
(b) final pH 7.2 ± 0.2

Levine Eosin Methylene Blue (EMB) agar

(a) autoclave at 121°C for 12-15 minutes or use boiling water bath or direct heat for dissolution
(b) final pH 7.1 ± 0.2

4.9 Standard Plate Count Agar

Temper melted agar (44° to 46°C) before pouring
Melted agar held no longer than eight hours
Do not melt sterile medium more than once
Autoclave at 121°C for 15 minutes, time adjusted depending on volume
Final pH 7.0 ± 0.2

Laboratory _____ Evaluator _____

Location _____ Date _____

5. Analytical Methodology

5.1 Use approved analytical methodology _____

5.2 Approval for tentative and alternate methods and other modifications received from the Alternate Test Procedure Program _____

5.3 Sample volume examined by MF is 100 mL ± 2.5 mL _____

5.4 Coliforms detected by MF procedure reported as coliform density per 100 mL _____

Confluent growth – membrane covered with bacterial growth _____
TNTC – greater than 200 total bacterial colonies _____

(a) if reported as confluent growth or TNTC with less than 5 coliforms, another sample requested from same sampling site _____
(b) if reported as confluent growth or TNTC with 5 or more coliforms, indicate an MCL violation and a check sample requested from same sampling site _____

5.5 Verification procedure conducted on all unsatisfactory samples _____

5.6 Completed MPN Test conducted on all unsatisfactory samples _____

QC 5.7 MF sterility check conducted at end of each filtration series _____

6. Sample Collection, Handling, and Preservation

6.1 Follow sample procedures described in Standard Methods and EPA Manual 600/8-78-017 _____

6.2 Sample collectors receive training _____

6.3 Samples representative of distribution system _____

6.4 Water taps free of any attachments and mixing type faucets _____

6.5 Water run to waste for at least two minutes _____

6.6 Sample volume is at least 100 mL with sufficient space for mixing sample _____

6.7 Sample report form completed by collector _____

6.8 Compliance with state chain-of-custody regulations, if required _____

6.9 Date and time of sample arrival at laboratory are recorded, date and time analysis begins are recorded _____

6.10 Transit time does not exceed 30 hours _____

If laboratory required by state regulation to examine samples after 30 hours and up to 48 hours, data are indicated as possibly invalid _____

All samples arriving in laboratory after 48 hours are rejected and new samples requested _____

Laboratory _____ Evaluator _____

Location _____ Date _____

7. Quality Assurance Program

7.1 Written QA plan implemented and available for review _____

7.2 Quality control records maintained for five years _____

QC 7.3 One quality control sample analyzed per year **(when available)** for parameter(s) measured _____

QC 7.4 Analyze one unknown performance sample satisfactorily per year **(when available)** for parameter(s) measured _____

8. Data Reporting

8.1 Data entered on the sample report form is checked and initialed _____

8.2 Sample report forms are retained by laboratory or State program for five years _____

9. Action Response by Laboratory

9.1 The proper authorities notified of unsatisfactory results _____

9.2 Proper authorities notified of check sample results _____

Chapter VI

Radiochemistry

Critical Elements for Certification

The technical criteria in this chapter are divided into two sections: Critical Elements for Certification and Recommended Practices. Only the first section will be used to determine certification status.

1. Personnel

Although there are no critical elements for laboratory personnel, laboratory administrators and evaluators should recognize training and experience as essential to the acquisition of valid compliance monitoring data. Recommended minimum standards can be found in the Recommended Practices section.

2. Laboratory Facilities

2.1 The analysis of compliance monitoring samples should be conducted in a laboratory facility where security and integrity of the drinking water samples and analytical data are provided. In addition, a work-place for wet chemistry operations and for equipment that is critical to valid measurement of radioactive contaminants is necessary.

2.2 The counting instrument(s) necessary for measurement of those radionuclides described in the NIPDWR must be located in a room other than the one in which samples and standards are being prepared and in which other types of wet chemical analyses are being performed. All instruments should be properly grounded, and a regulated power supply, either external or internal, should be available to each instrument.

2.3 In areas where radioactive standards are being prepared, care must be taken to minimize contamination of surfaces, other samples and personnel. Either bench surfaces of an impervious material covered with absorbent paper, or trays (stainless steel, plastic, or fiberglass) lined with absorbent paper are acceptable.

2.4 The following items are necessary in a laboratory performing even the most basic radiochemical measurements (gross alpha and gross beta radioactivities) for compliance monitoring of drinking water supplies.

2.4.1 Sink with tap water and connection to the sanitary sewer system.

2.4.2 Electrical outlets (120 VAC grounded).

2.4.3 Source of distilled or deionized water.

2.4.4 Exhaust hood.

2.4.5 For laboratories that are performing wet chemistry separations that require filtration of a precipitated fraction of the sample, a vacuum source (pump or aspirator) should also be available.

3. Laboratory Equipment and Supplies

The following equipment and supplies are necessary for the analyses of regulated radionuclides. If a laboratory is not to be certified for a particular radionuclide parameter, instruments specified for analysis of that parameter are not necessary.

3.1 General Instrumentation and Equipment:

3.1.1 Analytical balance: Precision, ± 0.05 mg. Minimum scale readability, 0.1 mg.

3.1.2 pH meter or specific ion meter:

3.1.2.1 pH meter: Accuracy, ± 0.5 units. Scale readability, ± 0.1 units.

3.1.2.2 Specific ion meter: Expanded scale millivolt capability. Readable and accurate to ± 0.1 mV.

3.1.3 Conductivity meter: Readable in ohms or mhos, a range of up to 2 million ohms or down to 0.5 micromhos ± 1 percent, and a sensitivity of 0.33 percent of full scale or better.

3.1.4 Drying oven or lamp: Gravity convection type, or infrared drying lamp.

3.1.5 Desiccator: Glass or plastic models, depending on particular application.

3.1.6 Hot Plate: Units with selectable temperature control for safe heating of laboratory reagents and samples.

3.1.7 Glassware: Borosilicate type glass. All volumetric glassware should be marked Class A, denoting that it meets Federal specifications and need not be calibrated before use.

3.1.8 Muffle furnace: Automatically controlled with a chamber capacity of at least 2,200 cc (10 × 9.5 × 23) and a maximum operating temperature of 1,000°C continuous and 1,100°C intermittent.

3.1.9 Centrifuge: General purpose table-top model with a maximum speed of at least 3,000 rpm and a loading option of 4 × 50 ml.

3.1.10 Fluorometer: Capable of detecting 0.0005 microgram of uranium.

3.2 Radiation Counting Instruments

The types of radiation counting systems needed to comply with measurements described in the NIPDWR, are set forth below.

3.2.1 Liquid scintillation system: A liquid scintillation system is essential if the laboratory is to be certified for the measurement of tritium in drinking water samples. The system must be such that the sensitivity will meet or exceed the requirements of section 141.25 of the NIPDWR.

3.2.2 Gas-flow proportional counting system: A gas-flow proportional counting system may be used for the measurement of gross alpha and gross beta activities, radium-226, radium-228, strontium-89, strontium-90, cesium-134, and iodine-131 as described in the reference cited in section 141.25(a). The detector may be either a "windowless" (internal proportional counter) or a "thin window" type. A combination of shielding and a cosmic (guard) detector operated in anticoincidence with the main detector must be used to achieve low background beta counting capability. The alpha and beta background count of the system must be such that the sensitivity of the radioanalysis of water samples will meet or exceed the requirement of section 141.25 of

the NIPDWR with reasonable counting time (not more than 1000 minutes).

3.2.3 Alpha scintillation counting system: For measurement of gross alpha activities and radium-226, a scintillation system designed for alpha counting may be substituted for the gas-flow proportional counter described. In such a system, a Mylar™ disc coated with a phosphor (silver-activated zinc sulfide) is either placed directly on the sample or on the face of a photomultiplier tube, enclosed within a light-tight container, along with the appropriate electronics (high voltage supply, preamplifier, amplifier, timer and scaler).

3.2.4 Low background alpha and beta counting systems other than a gas-flow proportional counting system: Such a system should have a cosmic guard detector operated in anti-coincidence with the signal from the sample detector, and shielding, such that the alpha background will not exceed 0.2 cpm and the beta background will not exceed 2.0 cpm for a 2 inch diameter counting planchet geometry.

3.2.5 Scintillation cell system: A scintillation system designed to accept scintillation flasks ("Lucas cells") should be used for the specific measurement of radium-226 by the radon emanation method. The system consists of a light-tight enclosure capable of accepting the scintillation flasks, a detector (phototube), and the appropriate electronics (high voltage supply, amplifier, timers, and scalers). The flasks (cells) needed for this measurement may either be purchased from commercial suppliers or constructed by the laboratory.

3.2.6 Gamma spectrometer systems: Either a sodium iodide, NaI(Tl), crystal; a solid state lithium drifted germanium detector; or a gamma-X photon detector connected to a Multichannel analyzer is required if the laboratory is to be certified for analyses of manmade photon emitters.

3.2.6.1 If a sodium iodide detector is used, a cylindrical 7.5 cm × 7.5 cm NaI crystal is satisfactory. However, a 10 cm × 10 cm crystal is recommended. The detector should be shielded with a minimum of 10 cm of iron or equivalent. It is recommended that the distance from the center of the detector to any part of the shield should not be less than 30 cm. The multichannel analyzer, in addition to appropriate electronics, should contain a memory of not less than 200 channels and at least one readout device.

3.2.6.2 A system with a lithium drifted germanium, or a high purity germanium, or a gamma-X photon detector may be used for measurement of manmade photon emitters if the efficiency of the detector is such that the sensitivity of the system meets the minimum detectable activity requirements cited in section 141.25 of the NIPDWR. These detectors should be shielded with a minimum of 10 cm of iron or equivalent. The multichannel analyzer, in addition to appropriate electronics, should contain a memory of not less than 2000 channels and at least one readout device.

4. General Laboratory Practices

Laboratory practices are specified in the Recommended Practices section.

5. Analytical Methodology

The approved methods indicated in the NIPDWR or U.S. EPA-approved alternate methods, are to be used for drinking water compliance monitoring.

6. Sample Collection, Handling, and Preservation

Table VI-1 gives critical elements for sample handing including Preservation, and Applicable Counting Instrumentation.

7. Quality Assurance

7.1 Quality control data and records must be available for inspection.

7.2 A laboratory must participate at least twice each year in those U.S. EPA laboratory intercomparison cross check studies that include each of the analyses for which the laboratory is, or wants to be, certified. Analytical results must be within control limits described in "Environmental Radioactivity Laboratory Intercomparison Studies Program-FY-1981-1982" (EPA-600/4-81-004), or in subsequent revisions.

7.3 A laboratory must also participate once each year in an appropriate water supply performance evaluation (blind sample) study administered by U.S. EPA. Analytical results must be within control limits established by U.S. EPA for each analysis for which the laboratory is, or wants to be, certified.

7.4 Operating manuals and calibration protocols for counting instruments should be available to analysts and technicians.

7.5 Calibration data and maintenance records on all radiation instruments and analytical balances should be maintained in a permanently bound record.

7.6 The following specifications are included in minimum daily quality control:

7.6.1 A minimum of 10-percent duplicate samples should be analyzed to verify internal laboratory precision for a specific analysis. The difference between duplicate measurements must be less than two times the stand-

Table VI-1

Parameter	Preservative[1]	Container[2]	Instrumentation[3]
Gross alpha	Conc. HCl or HNO_3 to pH < 2[4]	P or G	A, B or G
Gross beta	Conc. HCl or HNO_3 to pH < 2	P or G	A or G
Strontium-89	Conc. HCl or HNO_3 to pH < 2	P or G	A or G
Strontium-90	Conc. HCl or HNO_3 to pH < 2	P or G	A or G
Radium-226	Conc. HCl or HNO_3 to pH < 2	P or G	A, B, D or G
Radium-228	Conc. HCl or HNO_3 to pH < 2	P or G	A or G
Cesium-134	Conc. HCl to pH < 2	P or G	A, C or G
Iodine-131	None	P or G	A or G
Tritium	None	P or G	E
Uranium	Conc. HCl or HNO_3 to pH < 2	P or G	F
Photon emitters	Conc. HCl or HNO_3 to pH < 2	P or G	C

[1] It is recommended that the preservative be added to the sample at the time of collection unless suspended solids activity is to be measured. However, if the sample must be shipped to a laboratory or storage area, acidification of the sample (in its original container) may be delayed for a period not to exceed 5 days. A minimum of 16 hours must elapse between acidification and analysis.
[2] P = Plastic, hard or soft; G = Glass, hard or soft.
[3] A = Low background proportional system; B = Alpha scintillation system; C = Gamma spectrometer (NaI(Tl) or Ge(Li); D = Scintillation cell (radon) system; E = Liquid scintillation system (section C.2.a); F = Fluorometer (section C.1.i); G = Low background alpha and beta counting system other than gas-flow proportional.
[4] If HCl is used to acidify samples which are to be analyzed for gross alpha or gross beta activities, the acid salts must be converted to nitrate salts before transfer of the samples to planchets.

ard deviation of the specific analysis as described in EPA-600/4-81-004, Table 3. If difference exceeds two standard deviations, prior measurements are suspect, calculations and procedures should be examined and samples should be reanalyzed when necessary.

7.6.2 When 20 or more specific analyses are performed each day, a counting standard and a background sample should be measured with each 20 samples. If less than 20 specific analyses are performed in any 1 day, a counting standard and a background sample should be measured along with the samples.

7.7 Quality control performance charts, or performance records, should be maintained for each instrument.

7.8 The laboratory should prepare and follow a written QA plan (see Chapter III, section on QA plan).

8. Records and Data Reporting

8.1 Compliance monitoring activities should be made legally defensible by the records kept of such activities.

8.2 Records of radioanalyses for compliance monitoring of drinking water supplies must be kept by the laboratory for not less than three years. This includes raw data, calculations, quality control data, and reports.

8.3 Actual laboratory reports may be kept, or data may be transferred to tabular summaries provided that the following information is included:

8.3.1 Date, place, and time of sampling; name of person who collected the sample.

8.3.2 Identification of sample as to whether it is a routine distribution system sample, check sample, raw or process water sample, surface or ground water sample, or other special purpose sample.

8.3.3 Date of sample receipt and analysis.

8.3.4 Laboratory and persons responsible for performing analysis.

8.3.5 Analytical technique/method used.

8.3.6 Results of analysis.

9. Action Response to Laboratory Results

When action response is a designated laboratory responsibility, the proper authority must be promptly notified of non-compliance sample results, and a request must be made for resampling from the same sampling point.

The following items and procedures are considered to be good laboratory practices and are therefore recommended.

All laboratories are urged to maintain and continually improve their personnel, facilities, equipment, instrumentation, and quality control procedures. To ensure continued production of scientifically and legally defensible data, an ongoing training program should be an integral part of a laboratory's program.

1. Personnel

1.1 Analyst or technician responsible only for the measurement of gross alpha and gross beta radioactivities.

1.1.1 Academic training: Minimum of a high school diploma or its equivalent, plus at least 2 weeks of specialized training in standards and sample preparation, instrument calibration, calculations, and data handling.

1.1.2 Experience: Minimum of 6 months of on-the-job.

1.2 Analyst or technician responsible for the measurement of specific radionuclides described in the National Interim Primary Drinking Water Regulations.

1.2.1 Academic training: Minimum of bachelor's degree in chemistry, radiochemistry, radioisotope technology, or its equivalent.

1.2.2 Experience:[1] Minimum of 1 year of appropriate experience in radiation measurements and radiochemical procedures.

This analyst may be assisted in routine sample preparation and radioanalytical procedures by one or more technicians with at least the minimum qualifications described in item 1.1, provided that such work is directly supervised by the analyst.

1.3 Laboratory Supervisor, Manager, or Director.

1.3.1 Academic training: Minimum of bachelor's degree or its equivalent.

1.3.2 Experience:[1] Minimum of 5 years of experience.

2. Laboratory Facilities

2.1 Space

Laboratory space should be adequate (150 to 200 ft² per analyst is recommended). This space should contain no less than 15 linear ft of bench space.

2.2 Lighting

2.2.1 Lighting should be provided to wet-chemistry laboratory rooms to the extent of 100-150 foot candles.

2.2.2 70-100 footcandles is usually sufficient lighting for counting rooms. Controlled variable lighting is recommended for counting rooms in which liquid scintillation counting instruments are located.

2.3 Ventilation

2.3.1 An exhaust hood with a face velocity of 80-100 feet per minute and a maximum limit of 70 percent supplied outside air to the exhaust hood should effect an adequate ventilation in wet-chemistry laboratory rooms, if volatile chemicals are restricted to exhaust hood use.

2.3.2 For other rooms in the laboratory, including the counting rooms, where people are working routinely, a 20 percent or more air change per hour is recommended.

2.4 Temperature of this room should not exceed 26.7°C (80°F). Temperature variation under normal operating conditions should not exceed 5°C (9°F) per day.

3. Laboratory Equipment and Supplies

3.1 Safety cans and safety storage cabinets should be available and readily accessible to laboratory analysts for the safe use of volatile and flammable solvents.

3.2 Eye wash equipment and showers should also be readily accessible to laboratory personnel.

3.3 Fire protection and explosion protection equipment should also be readily available.

4. General Laboratory Practices

The following general laboratory practices are recommended:

4.1 Glassware preparation: All glassware should be washed in a warm detergent solution and thoroughly rinsed in tap water. A distilled water rinse should follow the tap water rinse. This cleaning procedure is sufficient for most analytical needs. However, specific analytical methods may dictate the need for more elaborate procedures for ensuring cleanliness of glassware, such as hot acid wash followed by tap water and distilled water rinses.

4.2 Water quality: All water used in preparation of reagents, standards, and samples should have resistance values

[1]Each year of college-level training in related scientific fields of demonstrated equivalency shall be considered equal to 1 year of work experience. Such a substitution should not exceed one-half of the required experience.

between 0.5 and 2.0 megohms (2.0 and 0.5 micromhos/cm) at 25°C. Excellent quality water has resistance values exceeding 1.0 megohms/cm (less than 1.0 micromhos/cm). If such high quality water is not available in the laboratory, it may be purchased from commercial suppliers; the laboratory should request a list of quality specifications for water purchased and periodically check actual quality against these specifications.

4.3 Chemicals and reagents: "Analytical reagent grade" (AR) chemicals should be used for most analyses. For more detailed information on reagent grades, consult the 14th edition of **Standard Methods for the Examination of Water and Wastewater.** Individual analytical procedures in **Standard Methods** often specify special requirements for reagents to be used.

4.4 Storage of radioactive standards and radioactive wastes: There should be an enclosed and properly labeled area, either within the analytical laboratory or in a separate room, for the safe storage (in suitable containers) of standards, samples, and radioactive wastes.

4.5 Standards and sample preparation: There should be a designated area within the laboratory for preparation of radioactive standards and samples. Adequate precautions should be taken in this area to ensure against radioactive contamination. Provisions should be made for safe storage and disposal of radioactive wastes and for monitoring of the work area.

4.6 High and Low Level Radioactive Material: Environmental samples (such as drinking water samples) that are to be analyzed for radioactivity content should be protected from other samples (and standard solutions) that contain significantly higher levels of radioactivity. Separate designated areas (separate rooms if possible) should be made available in the laboratory when routine samples of significantly different radioactivity levels are being brought into the laboratory.

5. Analytical Methodology

6. Sample Collection, Handling, and Preservation

7. Quality Assurance

The following practices are recommended:

7.1 A current service contract should be in effect on all balances.

7.2 Class S weights should be available to make periodic checks on balances and a record of such checks should be kept in a bound logbook.

7.3 Chemicals should be dated upon receipt of shipment and replaced when needed or before shelf life has been exceeded.

7.4 An electronics technician should be available or a service contract should be in effect for all major instruments.

8. Records and Data Reporting

9. Action Response to Laboratory Results

Sample Forms for On-Site Evaluation of Laboratories Involved in Analysis of Public Water Supplies—Radiochemistry

Laboratory _____

Street _____

City _____ State _____

Survey By _____

Affiliation _____

Date _____ Telephone No. _____

Laboratory _____ Evaluator _____

Location _____ Date _____

Personnel

Position/title	Name	Academic training				Present Specialty	Experience (years and area)
		HS	BA/BS	MA/MS	Ph.D.		
Analyst(s)/ technician(s)							
Supervisory analyst							
Laboratory supervisor/ director							
Support (e.g., electronic technician)							

Laboratory _____ Evaluator _____

Location _____ Date _____

Laboratory Facilities

Item	Available Yes	Available No	Comments
Laboratory Sink – with tap water and sanitary sewer connections			
Electrical outlets – 120V ac, grounded			
Distilled or deionized water			
Exhaust hood			
Vacuum source			
Counting Room Separate from wet chemistry, sample and standards preparation area			
Regulated power supply			
Adequate electrical ground			

Laboratory _____ Evaluator _____

Location _____ Date _____

General Laboratory Equipment and Instruments

tem	No. of Units	Manufacturer	Model	Age and Condition
Analytical balance				
pH meter				
Specific ion meter				
Conductivity meter				
Drying oven				
Infrared lamp				
Desiccator				
Hot plate				
Muffle furnace				
Centrifuge				
Fluorometer				

Laboratory _____ Date _____

Location _____ Evaluator _____

Thin Window Gas-Flow Proportional Counter

Instrument number	Manufacturer	Model	Year	Sample changing: Manual	Automatic	Capacity

	Counting gas	Window density (g/cm²)	Instrument background: Alpha Operating voltage	Alpha cpm	Beta Operating voltage	Beta cpm
Calibration Standard Type: Alpha Beta						

Supplier: Alpha Beta	Calibration frequency[1]: D	W	M	Other	Service Maintenance frequency[2]: Q	S	A	Other	Condition[3]: G	R	N

[1]Daily, weekly, monthly.
[2]Quarterly, semiannually, annually.
[3]Good operating but needs repair, not operating.

Are operating manuals readily available to the operator? Yes ☐ No ☐
Are calibration protocols available to the operator? Yes ☐ No ☐
Are calibrations kept in a permanent control chart record? Yes ☐ No ☐
Are permanent service maintenance records kept on these systems? Yes ☐ No ☐

Windowless Gas-Flow Proportional Counter

Instrument number	Manufacturer	Model	Year	Sample changing: Manual	Automatic	Capacity

	Counting gas	Sample dish diameter (in)	Instrument background: Alpha Operating voltage	Alpha cpm	Beta Operating voltage	Beta cpm
Calibration Standard Type: Alpha Beta						

Supplier: Alpha Beta	Calibration frequency[1]: D	W	M	Other	Service Maintenance frequency[2]: Q	S	A	Other	Condition[3]: G	R	N

[1]Daily, weekly, monthly.
[2]Quarterly, semiannually, annually.
[3]Good operating but needs repair, not operating.

Are operating manuals readily available to the operator? Yes ☐ No ☐
Are calibration protocols available to the operator? Yes ☐ No ☐
Are calibrations kept in a permanent control chart record? Yes ☐ No ☐
Are permanent service maintenance records kept on these systems? Yes ☐ No ☐

Laboratory _____ Date _____

Location _____ Evaluator _____

Low Background Alpha and Beta Counter (other than gas-flow proportional)

Instrument number	Manufacturer	Model	Year	Sample changing: Manual	Automatic	Capacity

	Sample dish diameter (in.)	Instrument background: Alpha Operating voltage	cpm	Beta Operating voltage	cpm	Gamma Operating voltage	cpm
Calibration Standard Type: Alpha Beta							

Supplier:	Calibration frequency[1]: D	W	M	Other	Service Maintenance frequency[2]: Q	S	A	Other	Condition[3]: G	R	N
Alpha											
Beta											

[1]Daily, weekly, monthly.
[2]Quarterly, semiannually, annually.
[3]Good operating but needs repair, not operating.

Are operating manuals readily available to the operator? Yes ☐ No ☐
Are calibration protocols available to the operator? Yes ☐ No ☐
Are calibrations kept in a permanent control chart record? Yes ☐ No ☐
Are permanent service maintenance records kept on these systems? Yes ☐ No ☐

Liquid Scintillation Counter

Instrument number	Manufacturer	Model	Year	Sample changing: Manual	Automatic	Capacity

	Discriminator channels: 1	2	3	Data readout: Visual	Channel printout: 1	2	3	External standard: Yes	No	Refrigeration: Yes	No
Calibration Standard Type:											

Supplier:	Calibration frequency[1]: D	W	M	Other	Service Maintenance frequency[2]: Q	S	A	Other	Condition[3]: G	R	N

[1]Daily, weekly, monthly.
[2]Quarterly, semiannually, annually.
[3]Good operating but needs repair, not operating.

Are operating manuals readily available to the operator? Yes ☐ No ☐
Are calibration protocols available to the operator? Yes ☐ No ☐
Are calibrations kept in a permanent control chart record? Yes ☐ No ☐
Are permanent service maintenance records kept on these systems? Yes ☐ No ☐

Laboratory _____ Date _____

Location _____ Evaluator _____

Alpha Scintillation Counter

Instrument number	Manufacturer	Model	Year	Sample changing: Manual	Automatic	Capacity

	Alpha phosphor location: Photo tube	Samples	Instrument background: Operating voltage	cpm
Calibration Standard Type:				

Supplier:	Calibration frequency[1]: D	W	M	Other	Service Maintenance frequency[2]: Q	S	A	Other	Condition[3]: G	R	N

[1]Daily, weekly, monthly.
[2]Quarterly, semiannually, annually.
[3]Good operating but needs repair, not operating.

Are operating manuals readily available to the operator? Yes ☐ No ☐
Are calibration protocols available to the operator? Yes ☐ No ☐
Are calibrations kept in a permanent control chart record? Yes ☐ No ☐
Are permanent service maintenance records kept on these systems? Yes ☐ No ☐

Radon Gas-Counting System

System number	Counting Instrument: Make	Model	Year	Gas-counting cells/system	Manufacturer of gas-counting cells

Calibration Standard Type:	Calibration frequency[1]: D	W	M	Other	Service Maintenance frequency[2]: Q	S	A	Other	Condition[3]: G	R	N
Supplier:											

[1]Daily, weekly, monthly.
[2]Quarterly, semiannually, annually.
[3]Good operating but needs repair, not operating.

Are operating manuals readily available to the operator? Yes ☐ No ☐
Are calibration protocols available to the operator? Yes ☐ No ☐
Are calibrations kept in a permanent control chart record? Yes ☐ No ☐
Are permanent service maintenance records kept on these systems? Yes ☐ No ☐

Laboratory _____ Date _____

Location _____ Evaluator _____

Gamma Spectrometer Systems

Detector System	Type	Make	System number / Model	Year	Size

	Analyzer System			
	Make	Model	Year	Channels
Calibration Standard Type:				

Supplier:	Calibration frequency[1]				Service Maintenance frequency[2]				Condition[3]		
	D	W	M	Other	Q	S	A	Other	G	R	N

[1]Daily, weekly, monthly.
[2]Quarterly, semiannually, annually.
[3]Good operating but needs repair, not operating.

Are operating manuals readily available to the operator? Yes □ No □
Are calibration protocols available to the operator? Yes □ No □
Are calibrations kept in a permanent control chart record? Yes □ No □
Are permanent service maintenance records kept on these systems? Yes □ No □

Laboratory _____ Date _____

Location _____ Evaluator _____

Sample Handling and Preservation

Parameter	Container Used	Preservative Used	Comments	Satisfactory Yes	Satisfactory No
Gross alpha activity					
Gross beta activity					
Strontium-89					
Strontium-90					
Radium-226					
Radium-228					
Cesium-134					
Iodine-131					
Tritium					
Uranium					
Photon emitters: a.					
b.					
c.					
d.					
e.					

Laboratory _____ Date _____

Location _____ Evaluator _____

Methodology

Parameter	Sample Load per Month	Method[1] used, Cite Edition, Year, and Page				[2]Approved Alternate	Other	Satisfactory	
		EPA	APHA	ASTM	HASL-300			Yes	No
Gross alpha activity									
Gross beta activity									
Strontium-89									
Strontium-90									
Radium-226									
Radium-228									
Cesium-134									
Iodine-131									
Tritium									
Uranium									
Photon emitters (identify): a.									
b.									
c.									
d.									
e.									

[1]Methods used, other than approved alternate methods, must be referenced in the Drinking Water Regulations (Federal Register)
[2]Cite approval date.

Laboratory _____ Date _____

Location _____ Evaluator _____

Quality Control

Item	Cross Check Studies (water)	A[1]	B[2]	Performance (Blind) Studies (Water)	A[1]	B[2]
Participation in intercomparison (cross check) studies and performance (blind) studies (studies conducted by EMSL-LV) Reporting Period: _____ To _____	Gross alpha			Gross alpha		
	Gross beta			Gross beta		
	Sr-89			Sr-89		
	Sr-90			Sr-90		
	Ra-226			Ra-226		
	Ra-228			Ra-228		
	Tritium			Uranium		
	Uranium			Cs-134		
	I-131			Cs-137		
	Cs-134			Co-60		
	Cs-137			Ru-106		
	Co-60			Written QA plan implemented and available for review		
	Ru-106					

	Yes	No	Frequency	Comments	Satisfactory Yes	Satisfactory No
Verification of sample results by duplicate sample analysis						
Use of quality control charts or records						
Calibration and maintenance records available						

[1]Scheduled frequency of participation by the Laboratory, times per year.
[2]Number of acceptable performances (results) in the past year, where an acceptable result is a normalized deviation from the known value of < 3.0 sigma.

Laboratory _____ Date _____

Location _____ Evaluator _____

Data Reporting

Item	Comments: system(s) used, frequency, etc.
Records kept for 3 years	
Actual laboratory reports	
Tabular summary	
Information included:	
Date	
Place of sampling	
Time of sampling	
Person collecting sample	
Date of receipt of sample	
Date of analysis	
Type of analysis	
Laboratory and person responsible	
Method(s) used	
Results	

Appendix A

Chain-of-Custody

A. Introduction

Written procedures for sample handling should be available and followed whenever samples are collected, transferred, stored, analyzed or destroyed. For the purposes of litigation, it is necessary to have an accurate written record which can be used to trace the possession and handling of samples from the moment of collection through analysis. The procedures defined here represent a means to satisfy this requirement.

A sample is in someone's "custody" if:

1. it is in one's actual physical possession;
2. it is in one's view, after being in one's physical possession;
3. it is in one's physical possession and then locked up so that no one can tamper with it;
4. it is kept in a secured area, restricted to authorized personnel only.

B. Sampling Collection, Handling and Identification

1. It is important that a minimum number of persons be involved in sample collection and handling. Guidelines established in standard manuals for sample collection, preservation and handling should be used. (e.g., EPA NPDES Compliance Sampling Inspection Manual, MCD 51; Standard Methods for Examination of Water and Wastewater). Field records should be completed at the time the sample is collected and should be signed or initialed, including the date and time, by the sample collector(s). Field records should contain the following information:

(a) unique sample or log number;
(b) date and time;
(c) source of sample (including name, location and sample type);
(d) preservative used;
(e) analyses required;
(f) name of collector(s);
(g) pertinent field data (pH, DO, Cl residual, etc.);
(h) serial number on seals and transportation cases.

2. Each sample is identified by affixing a pressure sensitive gummed label or standardized tag on the container(s). This label should contain the sample number, source of sample, preservative used, and the collector(s') initials. Analysis required should be identified. Where a label is not available, the same information should be written on the sample container with an indelible marking pen. An example of a sample identification tag is illustrated in Figure A-1.
3. The sample container should then be placed in a transportation case along with the chain-of-custody record form, pertinent field records, and analysis request form. The transportation case should then be sealed and labeled. All records should be filled out legibly in pen. The use of locked or sealed chests will eliminate the need for close control of individual sample containers. However, there will undoubtedly be occasions when the use of a chest will be inconvenient. On these occasions, the sampler should place a seal around the cap of the individual sample container which would indicate tampering if removed.

C. Transfer of Custody and Shipment

1. When transferring the possession of the samples, the transferee must sign and record the date and time on the chain-of-custody record. Custody transfers, if made to a sample custodian in the field, should account for each individual sample, although samples may be transferred as a group. Every person who takes custody must fill in the appropriate section of the chain-of-custody record.
2. The field custodian (or field sampler if a custodian has not been assigned) is responsible for properly packaging and dispatching samples to the appropriate laboratory for analysis. This responsibility includes filling out, dating, and signing the appropriate portion of the chain-of-custody record. A recommended chain-of-custody format is illustrated in Figure A-2.
3. All packages sent to the laboratory should be accompanied by the chain-of-custody record and other pertinent forms. A copy of these forms should be retained by the field custodian (either carbon or photocopy).
4. Mailed packages can be registered with return receipt requested. If packages are sent by common carrier, receipts should be retained as part of the permanent chain-of-custody documentation.
5. Samples to be transported must be packed to prevent breakage. If samples are shipped by mail or by other common carrier, the shipper must comply with any applicable Department of Transportation regulations. (Most water samples are exempt unless quantities of preservatives used are greater than certain levels.) The package must be sealed or locked to prevent tampering. Any evidence of tampering should be readily detected if adequate sealing devices are used.
6. If the field sampler delivers samples to the laboratory, custody may be relinquished to laboratory personnel. If appropriate personnel are not present to receive the samples, they should be locked in a designated area of the laboratory to prevent tampering. The person delivering the samples should make a log entry stating where and how the samples were delivered and secured. Laboratory personnel may then receive custody by noting in a logbook the absence of evidence of tampering, unlocking the secured area, and signing the custody sheet.

D. Laboratory Sample Control Procedures

Sample control procedures are necessary in the laboratory from the time of sample receipt to the time the sample is discarded. The following procedures are recommended for the laboratory:

1. A specific person must be designated custodian and an alternate designated to act as custodian in the custodian's absence. All incoming samples must be received by the custodian, who must indicate receipt by signing the accompanying custody/control forms and who must retain the signed forms as permanent records.
2. The custodian must maintain a permanent logbook to record, for each sample, the person delivering the sample, the person receiving the sample, date and time received, source of sample, date the sample was taken, sample identification or log number, how transmitted to the laboratory, and condition received (sealed, unsealed, broken container, or other pertinent remarks). This log should also show the movement of each sample within the laboratory; i.e., who removed the sample from the custody area, when it was removed, when it was returned, and when it was destroyed. A standardized format should be established for logbook entries.
3. A clean, dry, isolated room, building, and/or refrigerated space that can be securely locked from the outside must be designated as a "custody room."
4. The custodian must ensure that heat-sensitive samples, light-sensitive samples, radioactive samples, or other sample materials having unusual physical characteristics, or requiring special handling, are properly stored and maintained prior to analysis.
5. Distribution of samples to the analyst performing the analysis must be made by the custodian.
6. The laboratory area must be maintained as a secured area, restricted to authorized personnel only.

7. Laboratory personnel are responsible for the care and custody of the sample once it is received by them and must be prepared to testify that the sample was in their possession and view or secured in the laboratory at all times from the moment it was received from the custodian until the time that the analyses are completed.
8. Once the sample analyses are completed, the unused portion of the sample, together with all identifying labels, must be returned to the custodian. The returned tagged sample must be retained in the custody room until permission to destroy the sample is received by the custodian.
9. Samples will be destroyed only upon the order of the responsible laboratory official when it is certain that the information is no longer required or the samples have deteriorated. (For example, standard procedures should include discarding microbiological samples after the maximum holding time has elapsed.) The same procedure is true for sample tags. The logbook should show when each sample was discarded or if any sample tag was destroyed.
10. Procedures must be established for audits of sample control information. Records should be examined to determine traceability, completeness, and accuracy.

Figure A-1 Sample Identification Tag Examples

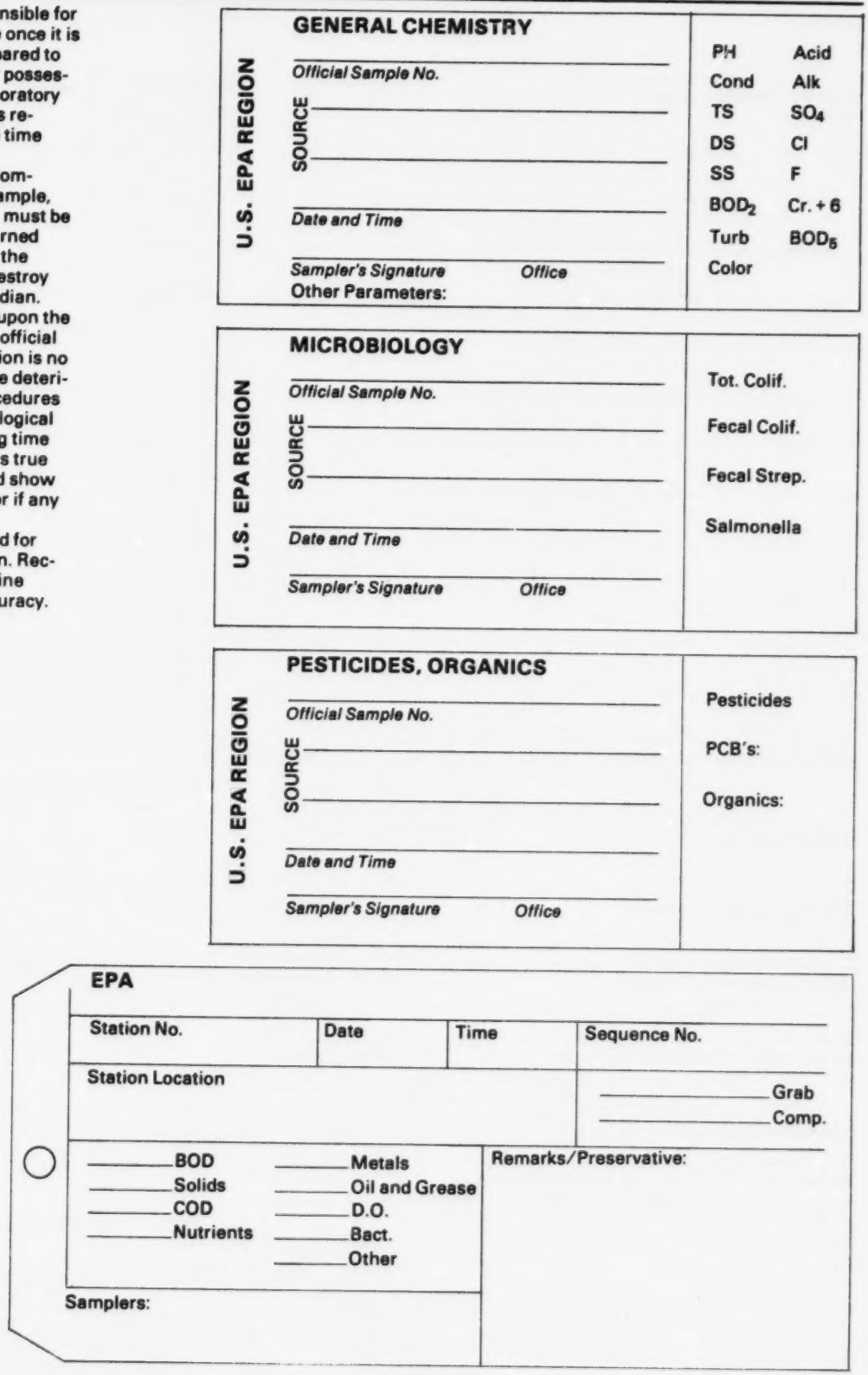

Figure A-2 Chain-of-Custody Record

Survey

Samplers: *Signature*

Station Number	Station Location	Date	Time	Sample Type: Water Comp.	Sample Type: Water Grab	Sample Type: Air	Seq. No.	No. of Containers	Analysis Required

Relinquished by: *Signature*	**Received by:** *Signature*	Date/Time
Relinquished by: *Signature*	**Received by:** *Signature*	Date/Time
Relinquished by: *Signature*	**Received by:** *Signature*	Date/Time
Relinquished by: *Signature*	**Received by Mobile Laboratory for Field analysis:** *Signature*	Date/Time

Dispatched by: *Signature*	Date/Time	Received for Laboratory by:	Date/Time

Method of Shipment:

Distribution: Orig.—Accompany Shipment
1 Copy—Survey Coordinator Field Files

Appendix B

Recommended Protocol for Regions Conducting On-Site Laboratory Evaluations

Before conducting the on-site evaluation, the Region shall:

- Hold a pre-evaluation conference with appropriate laboratory and field activity representatives to establish a schedule that would have a minimum impact on the laboratory activities.
- Request that a variety of tests be scheduled during the on-site evaluation.
- Arrange for the laboratory staff to be available during the on-site visit.

During the on-site visit, the team will:

- Evaluate the procedures and equipment used for those specific analyses for which the laboratory has requested certification, using the criteria in this manual.
- Review the records and written standard operating procedures for compliance with the required sampling frequency, sample collection, sample holding times, and if appropriate, resample notification.
- Insure that the laboratory has a QA plan in effect by:

— Determining if the laboratory has written procedures (QA plan or equivalent) for conducting its quality assurance program.
— Examining the quality assurance data to determine if the quality assurance program is being implemented.

- Complete the on-site checklists and other evaluation forms during the visit (see Chapters IV, V, and VI).
- Review the results of the evaluation with the director of the laboratory, the director of State water supply activities, and appropriate staff members. The review should:

— Discuss any deviations in the observed procedures and records.
— Recommend changes in equipment and supply needs, staffing requirements, and facility improvements, if necessary.
— Discuss possible assistance the Region can provide the laboratory.

Evaluation Report for Principal State Laboratories and Laboratories in Non-Primacy States

After an on-site inspection, the evaluation team should prepare a narrative report and action memorandum. This report should contain all information pertinent to the evaluation and also recommend the certification status for all analyses evaluated. The report should then be forwarded for evaluation to the Regional Director of the Environmental Services Division and the Regional Director of the Water Division. After considering the report, they should transmit it to the Regional Administrator for action.

The Regional Administrator should decide the certification status of the laboratory within 30 days and notify the State. The State should be sent the complete report. If the report indicates that the laboratory not be given Certified status for an analysis, the Regional Administrator shall give the specific reasons.

The narrative report should be attached to each copy of the completed evaluation form. It should include the general headings and information listed below:

Title Page

The title page should contain the following:

Report of an on-site evaluation of the
(name of laboratory)
at (city, State, and zip code)
on (date)
by
(name, title, organization,
and address
of the certification team)

Certification Status

List either Certified, Provisionally Certified, or Not Certified for each contaminant evaluated.

List of Deviations

List each deviation by item number used on the evaluation checklists. Describe the exact deviation and recommend changes.

Remarks

Recommend improvements which, while not affecting certification status, would improve laboratory operation. Other remarks might include reasons for failing the on-site evaluation, special recognition for outstanding performance, and description of unusual tests.

List of Personnel

List name and title of personnel along with the individual tests that each normally performs. Also identify the critical laboratory personnel.

Signature

Team members should sign the report.

Distribution

Copies of this report should be distributed to the State requesting the evaluation and EMSL-CI. For local laboratories in non-primacy States, reports should be distributed to appropriate Regional personnel.

Annually, each Region should submit to ODW a brief listing of laboratories in the Region having U.S. EPA or State certification status. The listing should include the names and location of each laboratory, and its certification status for all regulated contaminants. In addition, Regions should notify ODW of all changes in status soon after they occur so that ODW can maintain an updated list of certification status.

Appendix C

Abbreviations

ATP—Alternate analytical procedures (or techniques).

CA—Certifying authority. Regional Administrator for principal State laboratories and laboratories in non-primacy States; EMSL-CI and EMSL-LV for Regional laboratories.

DWLCI—Drinking Water Laboratory Certification Implementation Work Group.

EMSL-CI—Environmental Monitoring and Support Laboratory in Cincinnati, Ohio.

EMSL-LV—Environmental Monitoring Systems Laboratory in Las Vegas, Nevada.

MCL—Maximum Contaminant Level.

MERL—Municipal Environmental Research Laboratory in Cincinnati, Ohio.

NIPDWR—National Interim Primary Drinking Water Regulations.

ODW—Office of Drinking Water.

ORD—Office of Research and Development.

PE—Performance evaluation.

QA—Quality assurance.

QAMS—Quality Assurance Management Staff.

QC—Quality control.

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